UNIVERSIDADE FEDERAL DE LAVRAS

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RELEASE CHARACTERISTICS OF BLENDS FROM SLOW, CONTROLLED AND CONVENTIONAL NITROGEN FERTILIZERS AND UPTAKE BY CORN

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Dissertation presented to Soil Science Graduate Program of Federal University of Lavras, as part of the requirements of the Soil Science Graduate Program for the degree of Master of Science.

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ABSTRACT

Slow release fertilizers (SRF) and controlled release fertilizers (CRF) may improve nitrogen (N) use efficiency by crops, but their high costs are impeditive for widespread use. An alternative for that is the mixing of conventional fertilizer with SRF or CRF. The aim of this work was to assess the capacity of N sources in feed a corn crop in a pot experiment and to determine N release curves by polymer coated ureas at different aqueous mediums, by varying pH and ionic strength. The experiment was carried out under a greenhouse condition in pots filled with oxisol. The experimental design was a completely randomized factorial 14 x 4, with three repetitions. Treatments consisted of granular urea, ammonium nitrate, polymer coated ureas (multicote 4M®, urea + plastic resin and urea + polyurethane), urea formaldehyde and the mixing of CRF/SRF with granular urea (ratio of 40:60 in % of N and vice versa) in the rates of 0, 150, 300 and 450 mg N kg^{-1}. Three corn croppings were conducted, and at the end of each cropping, we evaluated nitrogen content, dry mass, nitrogen accumulation, SPAD index, agronomic efficiency index (AEI) and applied nitrogen recovery (ANR) in corn shoots after each cropping. The results varied widely among treatments given the wide range of N release capacity of the sources. For cumulative (sum of three croppings) dry mass, the order followed ammonium nitrate = M60:U40 = A40:U60 = urea + polyurethane > granular urea = multicote 4M® = M40:U60 = urea + plastic resin = A60:U40 = B40:U60 = B60:U40 = C40:U60 > urea formaldehyde = C60:U40. For cumulative N accumulation, the order followed ammonium nitrate > granular urea = urea + polyurethane = C40:U60 > M40:U60 = A40:U60 = A60:U40 = B40:U60 > multicote 4M® = M60:U40 = urea + plastic resin = B60:U40 > C60:U40 > urea formaldehyde. For the N release test, different aqueous mediums did not cause differences in N release at most of the days analysed. The use of blends is a promising option against high costs of CRF or SRF.

Keywords: Nitrogen. *Zea mays.* Fertilizer.

RESUMO

Os fertilizantes de liberação lenta (FLL) e os fertilizantes de liberação controlada (FLC) podem melhorar a eficiência de uso do nitrogênio (N) pelas culturas, mas seus altos custos são impeditivos para o uso generalizado. Uma alternativa para isso é a mistura de fertilizantes convencionais com FLL ou FLC. O objetivo deste trabalho foi de avaliar a capacidade de diferentes fontes nitrogenadas em nutrir a cultura do milho em um experimento de vaso e determinar curvas de liberação de N por ureias recobertas por polímero em diferentes meios aquosos, pela variação de pH e força iônica. O experimento foi conduzido sob condições de casa de vegetação em vasos enchidos com latossolo. O delineamento experimental foi um fatorial inteiramente casualizado 14 x 4, com três repetições. Os tratamentos consistiram de ureia granular, nitrato de amônio, ureias recobertas por polímeros (multicote 4M®, ureia + resina plástica e ureia + poliuretano), ureia formaldeído e a mistura de FLL/FLC com ureia granular (proporção de 40:60 em % de N e vice versa) nas doses de 0, 150, 300 e 450 mg N kg^{-1}. Três cultivos de milho foram conduzidos, e no final de cada cultivo, nós avaliamos o teor de N, massa seca, acúmulo de N, índice SPAD, índice de eficiência agronômica (AEI) e recuperação do N aplicado (ANR) na parte aérea de milho após cada cultivo. Os resultados variaram bastante entre tratamentos dado a ampla faixa de capacidade de liberação de N das fontes. Para o total (soma dos três cultivos) de massa seca, a ordem seguiu nitrato de amônio = M60:U40 = A40:U60 = ureia + poliuretano > ureia granular = multicote 4M® = M40:U60 = ureia + resina plástica = A60:U40 = B40:U60 = B60:U40 = C40:U60 > ureia formaldeído = C60:U40. Para o total de acúmulo de N, a ordem seguiu nitrato de amônio > ureia granular = ureia + poliuretano = C40:U60 > M40:U60 = A40:U60 = A60:U40 = B40:U60 > multicote 4M® = M60:U40 = ureia + resina plástica = B60:U40 > C60:U40 > ureia formaldeído. Para o teste de liberação de N, diferentes meios aquosos não causaram diferenças na liberação de N na maioria dos dias analisados. O uso de blends é uma opção promissora contra os altos custos dos FLC e FLL.

Palavras-chave: Nitrogênio. *Zea mays.* Fertilizante.

SUMMARY

1. INTRODUCTION

Nitrogen (N) is involved in reactions of plants and organisms, which makes it a nutrient quite uptake by plants (Cantarella, 2007). Plants use N coming from biological fixation, organic matter and electrical discharges, however, to reach competitive levels of yield of the crops it is indispensable the fertilization of N as mineral fertilizers, the main source of N of the conventional agriculture. N-mineral fertilizers are susceptible to losses by many process, however urea, the mineral fertilizer most widely used by agriculture in Brazil and world, stands for the large susceptibility of losses by volatilization, in case of an incorrect management of this fertilizer. According to (HUSSAIN; DEVI; MAJI, 2012), 40 to 70% of N applied by fertilizers are lost to the environment.

In order to target this problematic, slow and controlled release fertilizers are developed. They release the nutrient gradually, not immediately as urea, for example. However, a disadvantage of the improved N sources is the high cost. At this way, mix of enhanced efficiency fertilizers with conventional ones may proportionate a greater efficiency in the N-use, in a lower fertilization cost.

Another disadvantage of the slow and controlled release fertilizers is the unpredictability of the nutrient release, which certainly varies according to the fertilizer, climatic conditions and soil.

At this way, there is a need to find alternatives to reduce the fertilization cost, beyond methods that allow predicting the release rate of nutrient of slow and controlled release fertilizers.

The aim of this work was to assess dry mass, N content, N accumulation, SPAD index, agronomic efficiency index (AEI) and applied nitrogen recovery (ANR) of different N sources in corn.

2. LITERATURE REVIEW

2.1 Advantages and disadvantages of urea

About 78% of the atmosphere composition is nitrogen (N) (MARTINS et al., 2003). Plants may uptake N directly from atmosphere through biological nitrogen fixation and from the soils. In soils, most of N is in organic forms which must be mineralized to mineral forms of NH_4^+ and NO_3^- prior absorption. Only soil does not sustain profitable yields of crops (e.g. MARTINS, ISAAC SILVA; CAZETTA; FUKUDA, 2014), thus N should be supply through organic and/or (mainly) mineral fertilization. N is subjected to losses mainly by denitrification, ammonia (NH_3) volatilization, leaching and erosion (DE DATTA, 1981). Globally, losses of N through NH_3 volatilization was calculated to be 11 million tons of N per year, comprising 14% of the N fertilizers applied (BOUWMAN; BOUMANS; BATJES, 2002).

The ammonia volatilization causes acidification and eutrophication of ecosystems close to the emission source (SOMMER et al., 2004 cited by MARIANO et al., 2012). Many N mineral sources are available for the use in agriculture. Typical sources include urea, ammonium sulfate and ammonium nitrate. Although a wide variety of N sources, urea is the most traded source. In 2015, there was an apparent consumption of 4 127 059 tons of urea in Brazil, most of that amount imported, in contrast to 1 978 978 and 1 404 461 tons of ammonium sulfate and ammonium nitrate respectively (INTERNATIONAL PLANT NUTRITION INSTITUTE - IPNI, 2014). The reasons to this scenario are due to the technical-economic advantages related to this fertilizer such as: high N concentration, low production cost, transportation, storage and distribution, easy application without explosion risk, high water solubility, low corrosivity (KISS; SIMIHÃIAN, 2002), compatibility with many fertilizers (CIVARDI et al., 2011) and low acidifying capacity compared to other sources (MARCHESAN et al., 2011).

Due to the advantages of the use of urea, mainly by the high N concentration and hence low transportation cost, that is the preferable source by the landholders. However, urea presents disadvantages, such as possibility to NH_3 losses and biuret phytotoxicity (CANTARELLA, 2007).

The reaction of urea (CANCELLIER, 2013) in the soil is represented below:

$$(NH_2)_2CO + H_2O \xrightarrow{urease} NH_2COONH_4$$

$$NH_2COONH_4 + H_2O \rightarrow (NH_4)_2CO_3$$

$$(NH_4)_2CO_3 + 2\ H^+ \rightarrow 2\ NH_4^+ + CO_2 + H_2O$$

$$NH_4^+ + OH^- \leftrightarrow NH_3 + H_2O$$

$$CO_2 + H_2O \leftrightarrow H_2CO_3$$

$$H_2CO_3 \leftrightarrow HCO_3^- + H^+$$

Urease is an enzyme produced by bacterias, actinomycetes, soil fungi and sourced from plant debris (BYRNES, 2000).

When urea gets in touch with urease, the hydrolysis begins producing ammonium carbonate (COSTA; VITTI; CANTARELLA, 2003), followed by the others reactions described above. Urea is hydrolyzed in two or three days, and its rate is function of soil temperature, humidity, amount and the way the fertilizer is applied (BYRNES, 2000 cited by COSTA; VITTI; CANTARELLA, 2003).

The NH_3 loss is function of a range of factors, mainly by climatic conditions (e.g., wind speed, temperature, air relative humidity and rainfall), and by soil, for example, cation exchange capacity (CEC), humidity, temperature, organic matter content, nitrification potential, pH and NH_4^+ concentration in soil solution (ROCHETTE et al., 2009 cited by LORENSINI et al., 2012), gaseous exchange, water evaporation rate, clay content, buffer capacity (HARGROVE, 1988; BYRNES, 2000, cited by DUARTE et al., 2007).

Nitrogen losses may occur if pH is alkaline, low CEC and H buffer capacity, high temperature, low humidity and high N rates, or by the combination of one of the cited factors, or all of them (OLIVEIRA; BALBINO 1995 cited by CIVARDI et al., 2011).

For volatilization occurrence, it is required in the same time ammonium and high pH (SANGOI et al., 2003). The urea hydrolysis reaction increases the pH around the prill (ERNANI; STECKLING; BAYER, 2001), an undesirable feature (SCIVITTARO et al., 2010), and generates NH_3 and NH_4^+. That high pH condition makes difficult the transition of NH_3 to NH_4^+ due to lack of protons (H^+), causing NH_3 concentration close to urea prill with possibility to losses to atmosphere (VILLAS BÔAS et al., 2005)

The literature shows losses up to 78% of all N surface applied (LARA CABEZAS et al., 1997 cited by VILLAS BÔAS et al., 2005), but there are authors reporting values up to 94% (OLIVEIRA et al., 1997 cited by FARIA et al., 2013).

Thus, it is evident that strategies to mitigate volatilization of NH_3 should be applied to improve N uptake by crops.

2.2 Strategies for reducing losses and increase the N use efficiency

The incorporation of urea into the soil decreases ammonia volatilization, and is likely to be the simpler technique to the efficiency enhancement of that fertilizer. Cabezas, Trivelin and Pereira (2000) studying different N sources incorporated to the soil and applied in surface considered losses by NH_3 volatilization as ‘insignificants’ when sources were 5 to 7 cm incorporated. According to Lara Cabezas and Souza (2008) the efficiency of the incorporation of the urea is independent of soil type and climate conditions. The better use of N when incorporated is associated to two issues: 1) to the NH_4^+ favored adsorption to negative charges of the soil by the greater contact of the fertilizer to the soil (CUNHA et al., 2011), and 2) due to the greater distance between the fertilizer and the soil surface, the NH_3 formed reacts with H^+, becoming NH_4^+ as the gas rises to surface (SANGOI et al., 2003; CUNHA et al., 2011).

Although the urea incorporation be an effective practice against volatilization losses, the supply of all N required by a crop in the sowing phase is impossible, being required applying the fertilizer in topdressing (ESPINDULA et al., 2014). Furthermore, in the no-till system, there are landholders that use urea in topdressing (SANGOI et al., 2003), and its incorporation becomes a difficult task due to possible damage to roots. According to Faria et al. (2013), incorporation is an expensive practice, beyond dependent of the application rate, which leads to resistance of farmers for adoption. For incorporation under sugarcane straw, Mariano et al. (2012) agree that it is an expensive and low efficiency operation. For grasslands Oliveira, Trivelin and Oliveira (2007) found greater recovery of N-urea in shoots and accumulation in roots when urea was incorporated compared to topdressing application, however, that works with grasslands having a decumbent and stoloniferous growth, and not to cespitose grasslands due to difficulty in using implements agriculture according to the authors.

Another option to increase the efficiency of nitrogen fertilization is splitting the required dose, but that increases costs (RATKE et al., 2011). Indeed, there are others works studying losses by NH_3 volatilization as affected by diverse managements, for example, the supply of fertilizer at different time as in Da Ros, Aita and Giacomini. (2005) by applying urea in pre-sowing, sowing and topdressing in corn; urea applied at different soil humidity conditions under greenhouse (DUARTE et al., 2007), and mixing of urea and ammonium nitrate in solution (VITTI et al., 2007).

Although the knowledge that has been built over time, the development of technologies to increase the urea efficiency without incorporation in conservative systems is challenging (FARIA et al., 2013). Moreover, the yield of crops may be sustained applying about 70-80% of the actual rates of products traditionally used (BLAYLOCK, 2007), which motivates the studies to improve the use of nitrogen fertilizers.

2.3 Enhanced efficiency fertilizers

Enhanced efficiency fertilizers (EEFs) group three categories of fertilizers: stabilized fertilizers, slow-release (SRF) and controlled release fertilizers (CRF) (OZORES-HAMPTON; CARSON, 2013). Stabilized fertilizers are those which urease or nitrification inhibitors are associated to, and slow and controlled release fertilizers provide a release of the nutrient over time (TRENKEL, 2010).

To the American Association of Plant Food Control Officials (AAPFCO) the terms CRF and SRF do not mean different materials since the consider that no official differences between those types of fertilizers exist (CARSON; OZORES-HAMPTON, 2014). Thus, according to AAPFCO, CRF or SRF are fertilizers containing a plant nutrient that has its availability and use by plants delayed, or that has its availability made larger than a reference, such as urea (TRENKEL, 2010). According to Shaviv. 2005 cited by Trenkel (2010), CRFs are those which the rate, pattern and duration of the release are known and controllable during the CRF production. SRF still delays the nutrient availability, but that is not well controllable (TRENKEL, 2010). To Azeem et al. (2014), Trenkel, (2010) and Shaviv (2005) established differences between those materials: the nutrient release for SRF is almost unexpected and subjected to soil and climatic conditions changes while for CRF the release can be determined in a good sense. Others terms used as synonyms by the fertilizer industry are resin and polymer (ADAMS; FRANTZ; BUGBEE, 2013).

The definition of these categories of fertilizers is likely to be helpful to fertilizer market, while landholders may subscribe to new fertilizers products. The use of CRF is promising as much as more information is gathered about polymers (NI et al., 2011).

2.4 Advantages of SRF and CRF

According to Jin et al. (2013), the advantages of the use of SRFs are: reduction of losses, supply of nutrient of a sustainable manner, reduction of application frequency and diminishing possible negative effects caused by high rates of conventional fertilizers.

The use of SRF or CRF may provide advantages before and after the application in the field. One of the advantages of the coating of fertilizers is the less hygroscopicity (TIMILSENA et al., 2014), which make easier the storage and field management.

Based on a nutrient release over time, there is the possibility of performing applications at an unique rate (no splitting). According to Timilsena et al. (2014), conventional fertilizers may not supply a determined amount of nutrients to plants, reducing yield. Thus, fertilizers applied at greater rates in the beginning of crop growth is justified by the reduction of costs when fertilizers are split (TIMILSENA et al., 2014) with a lower environmental risk.

According to Adams, Frantz and Bugbee (2013), the supply of soluble fertilizers to soil normally causes higher nutrient concentration in soil solution which is greater than the ideal to plants, potentially susceptible to leaching losses and precipitation. Thus, the reduction of time in which the nutrient stays in solution may decrease the losses and increase the efficiency of use by plants

(GRANT et al., 2012). CRF or SRF optimize the ions concentration in soil solution during the growth period (ADAMS; FRANTZ; BUGBEE, 2013).

The low uptake of N by plants is partially due to the deficiency of synchronization between plant requirement and nutrient supply (LUPWAYI et al., 2010), with consequences in the agronomical and environmental efficiency (SHAVIV; RABAN; ZAIDEL, 2003)

According to Adams, Frantz and Bugbee (2013), an ideal CRF should balance its release to plant demand, even within an environmental variance. Common coating products are sulfur, wax, or plastic resin, or the arrangement of these products (GUERTAL, 2009).

The coating materials should be cheap and able to produce a good coating (HAN; CHEN; HU, 2009).

2.5 Mix of EEF with conventional urea

An interesting option against high costs of EEF is the mixture of conventional fertilizers with polymer-coated fertilizers (PCU). Noellsch et al. (2009) mixed urea and a CRF in the ration 1:1 by weight, and found a yield of 890 kg ha^{-1} greater compared to only urea applied at the year of 2006, but in 2005 urea solely applied showed 120 kg ha^{-1} greater yield than the mixture of the two fertilizers for the same position along a topossequence. The authors suggested this result was related to higher rainfall in 2006. In 2005, N recovery efficiency was 32 kg ha^{-1} for the blend at low-lying position and 19 kg ha^{-1} for urea treatment in the same position.

Villalba (2014) in two soils with clayey and medium textures and 30 and 12 g dm^{-3} of SOM content respectively, applied 180 kg ha^{-1} of N for an expected yield of 8-10 tons ha^{-1} of grains using conventional urea and PCU at ratios of 0, 50, 60,70,80,90 and 100% of the coated urea incorporated in the sowing, plus a control and split conventional urea treatments under conventional tillage. Control, blends and traditional N management were statistically similar for grain yield, shoot dry mass, N accumulation in shoot, N exported in grains and N use efficiency for the clayey soil. He speculated that no differences in yield was likely due to high mineralization potential of the soil and favorable environmental conditions and that a luxurious consumption of N could be occurred for N fertilized plots. To medium texture soil, among N fertilized plots, treatments using PCU showed higher yield compared to unique conventional urea and N split with conventional urea treatments. He found that mixing PCU and CU is efficient for corn crop but recommended more studies, especially in no-till system, stating that incorporation is not always a feasible practice. The author suggests that blends applied in surface should be investigated to find if they are efficient as well as when incorporated. He concludes that yield was affected by N fertilization only in the soil with medium texture, and the use of PCU resulted in higher yields compared to N split and all N applied at sowing when conventional urea is used.

2.6 Critical view on experiments assessing nitrogen fertilization in field and pot experiments

The published literature in Brazil has provided the scientific community with data regarding the use of different N fertilization managements, sources, rates and the effects on phytotechnical parameters and N nutrition on varying edaphoclimatic conditions. Among field experiments with corn crop, positive effects of the use of EEF sometimes is not observed. For instance, under ten years of no-till system and SOM content of 20.9 g kg^{-1} in Jataí (GO), Pereira et al. (2009) did not find differences in corn yield among control treatment, conventional urea applied at 40 and 80 kg N ha^{-1} and polymer-coated urea at 40 and 80 kg N ha^{-1} split twice as top-dressing. Urea with urease inhibitor at 80 kg N ha^{-1} yielded 7515 kg ha^{-1}, only statistically different to conventional urea at the lower rate of 40 kg N ha^{-1} and control plot, 6335 and 6298 kg ha^{-1}, respectively. It should be noted that 80 kg N ha^{-1} of stabilized urea was only 19% higher in yield than the control treatment. The lack of response among N fertilized plots may be due to the top-dressing N in twice, which likely increased the efficiency of conventional urea. Da Ros et al. (2015) suggest that splitting caused no differences of different N fertilizers as they did not show differences in N release rates.

Silva et al. (2012) in a soil with 22 g kg^{-1} of SOM content in Uberlândia (MG) did not find differences among conventional urea and two different coated urea applied in top-dressing to corn at 25 days after emergence under field conditions. Foliar N content, N in dry mass, production of dry and green mass, height of plants, stem diameter and yield were similar among N sources, although increasing rates affected positively vegetal characteristics and yields, showed by quadratic regressions. The absence of control treatment does not allow to infer about the natural capacity of soil to furnish N to crops by SOM mineralization. In the work of Mota et al. (2015) the control plot showed high capacity to provide mineral N and achieve a yield of 7.2 and 8 tons ha^{-1} in the agricultural years of 2011 and 2012, respectively.

Indeed, no positive effects over crops by using EEF are likely to occur under conditions that do not favor losses of N as NH_3 volatilization or N leaching (MOTA et al., 2015). That depends on several soil and climate conditions as well as the N source.

2.6.1 Water as a driving factor for EEF fertilization responses

Water is essential for crop growth, dissolution and incorporation of fertilizers, and nutrient uptake by plants since it is the medium that nutrients are dissolved in. For N, most of this nutrient is uptake by plants through mass flow, a transport that occurs with the movement of water.

If water is crucial for dissolution of fertilizers and consequently nutritional status of plants, its availability is certainly to affect crops by increasing or decreasing the amount of nutrients ready for plant uptake. Urea is a fertilizer tightly related to water conditions. At this view, water contributes for increasing N availability when 1) decreases NH_3 volatilization when urea is incorporated into soil by rainfall or irrigation, 2) decreases N leaching when rainfall is not enough for this and 3) determining soil humidity. It should be highlighted that mechanical incorporation of urea is also effective in reducing losses by volatilization.

Civardi et al. (2011) found a statistically greater profitability of incorporated conventional urea at a rate of 104,4 kg N ha^{-1} in the V5 stage of corn compared to PCU when surface applied at rates of 96.41 and 49.44 kg N ha^{-1} in a sandy soil in no-till system with 15.3 g dm-3 of SOM content. The yield was also statistically different among treatments, in the sequence incorporated conventional urea > controlled release fertilizer at the greater rate > polymer-coated urea at the minor rate. Incorporated urea caused higher ear length, ear diameter and grain hundred mass than polymer-coated urea surface applied.

Frazão et al. (2014) applied conventional urea, urea with urease inhibitor and coated urea under conventional tillage in a soil with SOM content of 28 g dm-3 in four rates top-dressed at V6 stage. Shoot dry mass and grain hundred mass did not differentiate among N sources (except for grain hundred mass variable, which N fertilized plots showed difference only to control treatment), but leaf N content and yield were affected by sources. Among PCU and conventional urea, N leaf content and yield showed statistically the same results. Shoot dry mass and grain hundred mass increased with rates regardless sources. Urea with urease inhibitor and PCU gave higher productivity compared to conventional urea at all rates, however, at the minor rate, this did not occur. N leaf concentration also increased in a quadratic form with rates at all treatments, but again, at the lower rate there was no differences among the sources tested. That is probably a consequence of SOM mineralization by masking the effect of N fertilization of the lower rate. Urease inhibitor urea showed higher yield compared to PCU calculated by quadratic regression, and the authors ascribed this result to the lack of rainfall during the first three days after fertilization, high temperatures and soil humidity. According to the authors, urea with urease inhibitor and PCU furnished adequate N nutrition only in the two highest rates and conventional urea caused reduction of yield and N deficiency. The authors conclude that the two enhanced efficiency urea help to supply N to plants.

Urea with urease inhibitor showed similar NH_3 losses when applied in moist soil (irrigation of 10 mm prior fertilization) and when irrigation was not done in the work of Viero et al. (2015) in the field. According to the authors, only urea with urease inhibitor lowered losses of NH_3 at the mentioned irrigation system compared to the others N sources. High temperature associated with moist soil was likely to favor NH_3 volatilization according to them. An overall view showed that all N sources provided reduced volatilization when irrigation was performed after fertilization, compared to no irrigation and irrigation prior fertilization. At the non-irrigation treatment, for cumulative NH_3 losses, conventional urea was similar to slow release urea. Urea with urease inhibitor reduced cumulative NH_3 volatilization by 57% compared to urea at no-irrigated plots. In irrigation prior fertilization, for cumulative losses, slow release urea did not differentiate to conventional urea but urea with urease inhibitor reduced by 37% compared to SRF and conventional urea. For irrigation after fertilization plots, slow release urea showed the greatest cumulative losses compared to urea with urease inhibitor and conventional urea. Cumulative losses from urea was reduced by irrigation after fertilization by 65%, for urea with urease inhibitor, 60% and 50% for slow release urea.

Noellsch et al. (2009) attributed higher corn yield and silage in 2006 compared to 2005 due to well distributed rainfall in that year. They conducted experiments at three positions at landscape: summit, sideslope and low-lying. PCU, conventional urea, mix of both and anhydrous ammonia were incorporated. A control plot was also considered. At the low position, soil remained almost wetter than the others position along the experiment conduction period. Grain yield of PCU and anhydrous ammonia were higher than conventional urea at the low position at 2005 and 2006. Mix of PCU and conventional urea showed higher yield at the middle position compared to conventional urea treatment in 2006 and the authors attributed this to higher rainfall at this year compared to 2005. Grain and silage in 2006 was greater than 2005 likely caused by well distributed rainfall, according to the authors.

Valderrama et al. (2014) in Selvíra (MS) under a soil with 53% of clay and 29 g dm^{-3} of SOM content tested conventional urea and three PCU. The area has been cultivated under no-till for four years. They applied the treatments at V6 stage and irrigated 14 mm to avoid volatilization at the seasons 2009/2010 and 2010 under irrigations when necessary. Sources did not differentiate regarding chlorophyll index and foliar N content at both experiments. Linear increase was found for foliar N content at both experiments. Significate increase with rates of foliar chlorophyll index for second crop and leaf N content for both crops followed linear regressions. Height of plants, height of first ear insertion and diameter at second internode were not affected by sources and rates. Yield at both crops was similar among sources although affected by rates at both cropping. The authors suggest that coating was not efficient due to the high temperatures of the study area. The maximum yield obtained was 15% and 23% greater than control treatment for first and second crop, respectively, according to the authors.

Martins, Cazetta and Fukuda (2014) under conventional tillage and SOM content of 25 g dm^{-3} in the upper layer of 20 cm, applied conventional urea and

PCU applying all rate as top-dressed and split, plus a control plot. Also, they tested the mentioned fertilizers with five rates applied at seven days after sowing. They assessed N-leaf content, dry mass of shoots, yield and a harvest index. They did not find differences among N sources for all variables analyzed. Splitting the rate showed 5% greater of yield compared to all rate applied at 7 days after sowing. They suggest that high temperatures and intense rainfall favored corn response to splitting fertilization against the rate fully applied once. They did not find differences among N sources and control plot for all variables, except for yield, and authors state that soil had N in levels enough to cause adequate N content in leaves, but not enough for sustain high yields. N fertilization on yield did not reach the expected 10-12 tons and they ascribed this to high temperatures and intense rainfall. They speculated that low yield response could have ameliorated differences among treatments. In the experiment assessing five rates, they found greater values in shoots-dry mass, yield and harvest index when PCU was used, and no differences among sources for N-leaf content and grain hundred mass. According to the authors, higher yield and shoot dry mass were likely due to the lack of significate rainfall, which posed soil humidity increasing losses by conventional urea, but not enough for dissolution of PCU and thus lower losses of the controlled fertilizer. Rates of N affected yield and hundred grain mass. PCU caused linear increases of yield along rates and conventional urea did not respond to rates. The authors suggest that both urea proportionate similar losses, but greater results obtained from PCU in rates greater than 170 kg ha^{-1} of N show that there is possibility for advantages when using PCU. The authors suggest that PCU is likely to show similar responses compared to conventional urea when applied under irrigated soils or under rainy periods. In the microscope, the authors observed that PCU and conventional urea dissolved at the same time in the first 4.5 minutes in distilled water, and they suggested that under dry conditions polymer protects urea but under a rainfall event or irrigation that causes soil soaking, within some minutes all the urea content would be equally dissolved compared to conventional urea, and thus, both fertilizers exposed to the same conditions causing similar responses to crops. Through their observation in the microscope, authors suggest that inconsistent results when assessing PCF (polymer coated fertilizer) may be ascribed to soil and climate conditions before and along the fertilization. According to them, such details are not always well assessed in front of illogical results in the works. They speculate that PCU is not advantageous under rainy season and irrigation system.

EEF have also been assessed under greenhouse conditions and are likely to represent a harsher condition compared to field trials since they depend of daily watering. Rampim et al. (2010) did not find differences regarding N content and dry mass when urea, urea with urease inhibitor, mix of ammonium sulfate and urea, ammonium sulfate and urea mixed with oil was applied in a sandy soil with 13.67 g dm^{-3} of SOM content in a greenhouse experiment. According to the authors, frequent pot irrigations cause no differences to corn crop regarding N use. In fact, irrigation is an important aspect to greenhouse experiments dealing with N fertilization. In a field trial, rainfall may not occur in the subsequent days of fertilization, thus fertilizers are not incorporated. In a pot experiment, daily irrigations are necessary since plants are affected by soil water content due to low soil volume for roots. Silva et al. (2012) in a soil with 63% of sand and 21 mg dm^{-3} of SOM content did not find differences in N content in dry mass when monoammonium phosphate (MAP) and two polymered MAP were applied. By the other hand, accumulation of N in dry mass was affected by rates of the fertilizers.

2.6.2 SOM as a possible driving effect for non-responsive EEF fertilization

SOM contributes to N nutrition of plants as N-organic compounds are mineralized to mineral forms of NH_4^+ firstly, and NO_3^- if conditions are propitious for nitrification. SOM should be accounted for N management as well as any others N-credits supplied for plants as organic fertilizers and electrical discharge for example.

Measuring the role of SOM to N nutrition of plants is a hard task since that is a phenomenon tightly related to biological factors as well as climate conditions. That certainly implies in difficulties to establish an extractor to determine the amount of N that a soil is able to provide. Because of that, few works have considered the SOM content on their discussions in an attempt to explain the lack of differences among different N treatments in respect to the variables measured. Because of that, those works are only of speculative character.

Zavaschi et al. (2014) studied N fertilization with conventional urea and PCU surface applied under an area of 18 years of no-till system and 29 g dm^{-3} in the 0-10 cm depth. PCU did not cause statistically increase on corn yield, N leaf content, grain N concentrations and SPAD readings compared to conventional urea. Although N fertilization promoted numerical increases on yield, they did not differentiate of the control treatment. The authors attributed this event due to mineralization of N-organic matter and the stimulating effect of N fertilization upon mineralization, so-called ‘priming effect’. Due to maize-soybean rotation at the studied area, the authors state that N-soil stocks are notable. The authors concluded that no differences on N leaf and SPAD reading within fertilized plots are due to the main source of N supplied to corn crop in the experiment, the SOM mineralization.

Qiu et al. (2016) calculated the priming effect of N and residues applied to soil. They sampled a soil at 0-20 cm depth with 9.09 g kg^{-1} of SOC content in China. They incubated soil with ^{15}N-urea and ^{13}C provided by maize residues along 250 days. The authors quantified the priming effect indirectly through emissions of CO_2 evolved by the following treatments: maize residues applied to soil, urea and combination of residues plus urea, more one control treatment. The findings were: cumulative CO_2 emissions of 9% greater for N addition treatment compared to control, a stimulation of native SOC decomposition of 9.1%, called by the authors as positive priming effect. The addition of N caused mineralization of maize residues after 20 days of incubation. Maize plus N increased mineral N availability. Microbial biomass C was increased by 84, 59 and 6% when maize, maize plus N and N were added to soil, respectively. Dissolved organic carbon concentration increased by 19, 10 and 5%, respectively for maize, maize plus N and N treatments, compared to control.

Indeed, the successful of EEF on N nutrition is certainly associated to one or more factors acting together on avoiding losses. Mota et al. (2015) applied at V6 stage ammonium nitrate, conventional urea, urea with urease inhibitor and urea with nitrification inhibitor at four rates under an area with a historic of twelve years of no-till system and 50 g kg^{-1} of SOM content at upper 20 cm layer and 42% of clay. The authors conducted the same experiment during two growing seasons and did not find differences among N fertilizers regarding N use agronomic efficiency, yield, foliar N content and relative chlorophyll content for both years. For these three last variables, linear increases occurred with increasing rates. For the first year, thousand-kernel weight did differ among sources. N use efficiency was affected by rates in the first year, being the lower value at the maximum rate. In the second cropping, N use efficiency was not affected by rates. The authors attribute unresponsive differences of some variables due to conditions that did not allow losses of N. In both cropping year, they speculate that most corn N uptake occurred after fertilization but prior considerable rainfall in the twelfth day. In addition they suggest that the low pH of 5.6, high SOM content of 50 g kg^{-1}, fertilizer application at surface, fine texture and well distributed rainfall did avoid N leaching, and low pH, high CEC of 15.3 cmolc kg^{-1}, mild temperatures of 18.8 up to 21.4 C, low soil humidity at fertilization day which occurred after five days of the last rainfall, possible rainfall incorporation but not causing leaching (27 mm) at the first cropping and 28.5 mm from second to seventh day after fertilization during a great N requirement by plants. In addition, pre cropping in the middle of the year, consolidate no-till, high SOM content may help to do not differentiate sources regarding yield according to the authors.

Zhao et al. (2013) in a soil with 19.7 g kg^{-1} of SOM content applied two NPK-coated fertilizers (resin and sulfur coatings) corresponding to 50, 75 and 100% of the rate used for conventional fertilizer treatment in corn crop as a basal fertilization. Yield was higher for coated-fertilizers applied at rates of 75% and 100% compared to conventional fertilizer. For 50% treatments, these were similar to conventional fertilization. Among the two coated fertilizers, there was no difference. Net photosynthetic rate was similar among treatments, but the decreased of this variable after flowering was slowed down for 75 and 100% treatments for both coated fertilizers and the authors attributed the higher yield to the higher photosynthetic rates. Fluxes of NH_3 were lowered to all coated fertilizers compared to conventional and peaks were postponed for coated fertilizers. Use of coated fertilizers delayed senescence of plants. Agronomic N use efficiency and N recovery were greater for resin coated fertilizer than conventional fertilization. The authors found greater residual N in the 0-100 cm depth for resin-coated fertilizer than conventional treatment.

In field experiments when N treatments are applied as top-dressing fertilization, base fertilization with N is a common activity and needful, which certainly contributes for no differences as N is a mobile nutrient in plants and base fertilization is partially responsible for sustaining N requirements up to stage that topdressing fertilization is applied, ranging from v4 to v6 stages for corn crop.

The ^{15}N technique is a valuable tool that enable to quantify the amount of N furnished by a fertilizer to crops, however, it is an expensive attempt. A cheaper approach would be the assessment of N nutrition in a free-soil organic matter soil, but that is impossible for field experiments. By the other hand, pot experiments represent an appropriate opportunity for studying N fertilization and uptaking by plants through the use of a substrate free of soil organic matter. A free-organic matter soil is impossible but a better approach would be the use of subsurface soil whether sampled in deeper layers, thus low or negligible soil organic matter.

To the best of knowledge, there is no experiment that has target such scenario. To study crop N nutrition by EEF under conservative systems are valuable in order to detect the feasibility of this technology under such conditions, however due to the soil organic matter role, differences among a range of N fertilizers may not be detected. That could terminate in misleading conclusions regarding the release capacity and thus functionality of EEF. Therefore, research upon N furnishment by fertilizers are not available, and thus, a gap at this topic should be target.

2.7 Release test of CRF

The release of nutrients may be performed under laboratory or field conditions, whether it is done in field, environmental conditions cannot be controlled, while under laboratory conditions such variables may be controlled and adjusted to optimum values (MEDINA et al., 2014), which certainly diminish or increase the release. Although the valuable assessment of the nutrient release in the field, there is a need to stablish a methodology in a lab able to predict the N release (MEDINA et al., 2014).

Release tests may be accomplished in aqueous or solid medium. However, in solid medium, potential chemical effects sometimes are not taken into account in the release tests (ADAMS; FRANTZ; BUGBEE, 2013). When done in water, the time required for a release test under 25 °C is too long for tests in commercial products and high temperatures may cause breakups of the membrane (DAI et al., 2008).

Although release tests from CRF be investigated since the 50's (DAI et al., 2008), a methodology still is necessary to estimate the nutrient release of broad range of materials (MEDINA; SARTAIN; OBREZA, 2009). There is no standard method to characterize the nutrient release of CRFs accepted by the Association of Official Analytical Chemists and, among several technologies, none have evaluated in a precise manner the longevity of a wide range of materials (MEDINA et al., 2014), being the subject of efforts by researchers for a worldwide method (OZORES-HAMPTON; CARSON, 2013).

The standard of nutrient release stated by manufacturers, in lab or greenhouse conditions, sometimes does not match to its performance in the field (MEDINA et al., 2008). However, most of the release tests are done in distilled or deionized water, thus ignoring ions in the soil and its pH. For example, Liang and Liu (2006) tested the release of N by urea coated by them in aqueous medium with different pH values and in soil. These authors found lower release rates in soil, and suggest that ions in soil affected the coating of the fertilizer produced. According to Medina et al. (2008), knowing the release time and features of each CRF is needed to increase its use by landholders.

According to Trenkel (2010), the methods should meet some criteria, such as: possibility to be done in an analytical lab, may be done in seven days or less, easy operation with available materials, applicable to a wide range of materials, able to match with data in the field, and others.

N release of coated products may be dependent of humidity and soil temperature, microbial activity, coating thickness, size of the hole in the coating, or the mix of some of the mentioned variables (GUERTAL, 2009), being temperature the more preponderant among the external factors (ADAMS; FRANTZ; BUGBEE, 2013). According to these authors, there is no agreement regarding the temperature effect over polymer-coated fertilizers, having wide variation in the release rates depending on fertilizer and nutrient. The release of nutrient of each prill in a population is not similar due to differences in the coating thickness, breakups of the coating, residual humidity content and response of the coating material to environmental conditions, beyond the age of the fertilizer (TIMILSENA et al., 2014). According to these authors, features of the material used to cover are as important as the external factors in the nutrient release.

Coating materials and manufacturing processes of the fertilizers have been developed by many companies to each type of fertilizer coated with polymers (ADAMS; FRANTZ; BUGBEE, 2013). The nutrient release is dependent of several external factors and of the fertilizer (AZEEM et al., 2014), beyond the possibility of many mechanisms acting together (ADAMS; FRANTZ; BUGBEE, 2013). The variety of materials available, in addition to several variables responsible for the nutrient release certainly complicates the studies of release and its predictability. The difficulty on foresee the N release is one of the negative features of the use of such fertilizers, in addition to the higher price and lack of rapid response in the plant growth (GUERTAL, 2009).

The objective of this work was to assess the capacity of N sources in feed a corn crop in a pot experiment with a soil poor in organic matter and to determine N release curves by polymer coated urea.

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Chapter 1 RELEASE CHARACTERISTICS OF BLENDS FROM SLOW, CONTROLLED AND CONVENTIONAL NITROGEN FERTILIZERS AND UPTAKE BY CORN

ABSTRACT

Slow release fertilizer (SRF) and controlled release fertilizer (CRF) may improve nitrogen (N) use efficiency by crops, but their high costs are impeditive for widespread use. An alternative for that is the mixing of conventional fertilizer with SRF or CRF. The aim of this work was to assess the capacity of N sources in feed a corn crop in a pot experiment and to determine N release curves by polymer coated ureas at different aqueous mediums, by varying pH and ionic strength. The experiment was carried out under a greenhouse condition in pots filled with oxisol. The experimental design was a completely randomized factorial 14 x 4, with three repetitions. Treatments consisted of granular urea, ammonium nitrate, polymer coated ureas (multicote 4M®, urea + plastic resin and urea + polyurethane), urea formaldehyde and the mixing of CRF/SRF with granular urea (ratio of 40:60 in % of N and vice versa) in the rates of 0, 150, 300 and 450 mg N kg^{-1}. Three corn croppings were conducted, and at the end of each cropping, we evaluated nitrogen content, dry mass, nitrogen accumulation, SPAD index, agronomic efficiency index (AEI) and applied nitrogen recovery (ANR) in corn shoots after each cropping. The results varied widely among treatments given the wide range of N release capacity of the sources. For cumulative (sum of three croppings) dry mass, the order followed ammonium nitrate = M60:U40 = A40:U60 = urea + polyurethane > granular urea = multicote 4M® = M40:U60 = urea + plastic resin = A60:U40 = B40:U60 = B60:U40 = C40:U60 > urea formaldehyde = C60:U40. For cumulative N accumulation, the order followed ammonium nitrate > granular urea = urea + polyurethane = C40:U60 > M40:U60 = A40:U60 = A60:U40 = B40:U60 > multicote 4M® = M60:U40 = urea + plastic resin = B60:U40 > C60:U40 > urea formaldehyde. For the N release test, different aqueous mediums did not cause differences in N release at most of the days analysed. The use of blends is a promising option against high costs of CRF or SRF.

Keywords: Nitrogen. *Zea mays.* Fertilizer.

RESUMO

Os fertilizantes de liberação lenta (FLL) e os fertilizantes de liberação controlada (FLC) podem melhorar a eficiência de uso do nitrogênio (N) pelas culturas, mas seus altos custos são impeditivos para o uso generalizado. Uma alternativa para isso é a mistura de fertilizantes convencionais com FLL ou FLC. O objetivo deste trabalho foi de avaliar a capacidade de diferentes fontes nitrogenadas em nutrir a cultura do milho em um experimento de vaso e determinar curvas de liberação de N por ureias recobertas por polímero em diferentes meios aquosos, pela variação de pH e força iônica. O experimento foi conduzido sob condições de casa de vegetação em vasos enchidos com latossolo. O delineamento experimental foi um fatorial inteiramente casualizado 14 x 4, com três repetições. Os tratamentos consistiram de ureia granular, nitrato de amônio, ureias recobertas por polímeros (multicote 4M®, ureia + resina plástica e ureia + poliuretano), ureia formaldeído e a mistura de FLL/FLC com ureia granular (proporção de 40:60 em % de N e vice versa) nas doses de 0, 150, 300 e 450 mg N kg^{-1}. Três cultivos de milho foram conduzidos, e no final de cada cultivo, nós avaliamos o teor de N, massa seca, acúmulo de N, índice SPAD, índice de eficiência agronômica (AEI) e recuperação do N aplicado (ANR) na parte aérea de milho após cada cultivo. Os resultados variaram bastante entre tratamentos dado a ampla faixa de capacidade de liberação de N das fontes. Para o total (soma dos três cultivos) de massa seca, a ordem seguiu nitrato de amônio = M60:U40 = A40:U60 = ureia + poliuretano > ureia granular = multicote 4M® = M40:U60 = ureia + resina plástica = A60:U40 = B40:U60 = B60:U40 = C40:U60 > ureia formaldeído = C60:U40. Para o total de acúmulo de N, a ordem seguiu nitrato de amônio > ureia granular = urea + poliuretano = C40:U60 > M40:U60 = A40:U60 = A60:U40 = B40:U60 > multicote 4M® = M60:U40 = ureia + resina plástica = B60:U40 > C60:U40 > ureia formaldeído. Para o teste de liberação de N, diferentes meios aquosos não causaram diferenças na liberação de N na maioria dos dias analisados. O uso de blends é uma opção promissora contra os altos custos dos FLC e FLL.

Palavras-chave: Nitrogênio. *Zea mays*. Fertilizante.

1 INTRODUCTION

Mineral nitrogen fertilization is a common agricultural practice applied by landholders to ensure profitable yields. Only soil does not sustain profitable yields of crops, thus N should be supply through mineral fertilization (e.g. MARTINS; CAZETTA; FUKUDA, 2014). Thus, the risk of excess N fertilization is relevant.

Urea is the main mineral N fertilizer applied to crops. However, it is susceptible to losses as NH_3 volatilization. Globally, losses of N through NH_3 volatilization was calculated to be 11 million tons of N per year, comprising 14% of the N fertilizers applied (BOUWMAN; BOUMANS; BATJES, 2002). The urea hydrolysis reaction increases the pH around the prill (ERNANI; STECKLING; BAYER, 2001), due to H^+ consumption by the reaction. That high pH condition makes difficult the transition of NH_3 to NH4 due to lack of protons (H^+), causing NH_3 concentration close to urea prill with possibility to losses to atmosphere (VILLAS BÔAS et al., 2005).

The incorporation of urea into the soil decreases ammonia volatilization, and is likely to be the simpler technique to the efficiency enhancement of that fertilizer. However, the supply of all N required by a crop in the sowing phase is impossible (ESPINDULA et al., 2014).

Another option to increase the efficiency of nitrogen fertilization is splitting the required dose, but that increases costs (RATKE et al., 2011). Although the knowledge that has been built over time, the development of technologies to increase the urea efficiency without incorporation in conservative systems is challenging (FARIA et al., 2013). Moreover, the yield of crops may be sustained applying about 70-80% of the actual rates of products traditionally used (BLAYLOCK, 2007), which motivates the studies to improve the use of nitrogen fertilizers.

Enhanced efficiency fertilizers (EEFs) group three categories of fertilizers: stabilized fertilizers, slow-release (SRF) and controlled release fertilizers (CRF) (OZORES-HAMPTON; CARSON, 2013). Stabilized fertilizers are those which urease or nitrification inhibitors are associated to, and slow and controlled release fertilizers provide a release of the nutrient over time (TRENKEL, 2010). One of the disadvantages of EEFs are their higher costs compared to conventional fertilizers.

An interesting option against high costs of EEF is the mixture of conventional fertilizers with polymer-coated fertilizers (PCU). Noellsch et al. (2009) mixed urea and a CRF in the ratio 1:1, and found a yield of 890 kg ha^{-1} greater compared to only conventional urea applied in 2006, but in 2005 that did not occur for the same area. Villalba, (2014) found that mixing PCU and CU incorporated into soil is efficient for corn crop but recommended more studies, especially in no-till system, stating that incorporation is not always a feasible practice.

The use of EEF is thought to be advantageous compared to conventional sources. However, no positive effects over crops by using EEF are likely to occur under conditions that do not favor losses of N as NH3 volatilization or N leaching (MOTA et al., 2015) when conventional fertilizers are used. For example, Pereira et al. (2009) did not find differences in corn yield among control treatment, conventional urea and polymer-coated urea split twice as top-dressing. Da Ros et al. (2015) suggest that splitting caused no differences of different N fertilizers as they did not show differences in N release rates. Civardi et al. (2011) found greater profitability of incorporated conventional urea (which diminishes NH_3 volatilization) at a rate of 104,4 kg N ha^{-1} compared to PCU when surface applied at rates of 96.41 and 49.44 kg N ha^{-1} in a sandy soil in no-till system. Frazão et al. (2014) found that urease inhibitor urea showed higher yield compared to PCU and ascribed this result to the lack of rainfall during the first three days after fertilization, high temperatures and soil humidity. In the work of Viero et al. (2015), only urea with urease inhibitor lowered losses of NH_3 when applied to moist soil compared to conventional urea and slow release urea. Cumulative losses from urea was reduced by irrigation after fertilization by 65%, for urea with urease inhibitor, 60% and 50% for slow release urea according to the authors. Valderrama et al. (2014) found no differences regarding corn yield in two growing season when testing conventional urea and three polymer coated urea under an irrigation system. Martins, Cazetta and Fukuda (2014) tested conventional urea and a PCU applying all rate as top-dressed and split, plus a control plot. They did not find differences among N sources and control plot for all variables, except for yield, and authors state that soil had N in levels enough to cause adequate N content in leaves, but not enough for sustain high yields. The authors suggest that PCU is likely to show similar responses compared to conventional urea when applied under irrigated soils or under rainy periods.

In the literature, few works have investigated N fertilization in corn under greenhouse conditions. Moreover, to the best of our knowledge there is no reports targeting the capacity of different EEF on providing N along cropping cycles. At pot experiment, Rampim et al. (2010) did not find differences regarding N content and dry mass when urea, urea with urease inhibitor, mix of ammonium sulfate and urea, ammonium sulfate and urea mixed with oil was applied in a sandy soil. According to the authors, frequent pot irrigations cause no differences to corn crop regarding N use. Silva et al. (2012) did not find differences in N content in dry mass when monoammonium phosphate (MAP) and two polymered MAP were applied.

The objective of this work was to assess the capacity of N sources in feed a corn crop in a pot experiment with a soil poor in organic matter and to determine N release curves by polymer coated urea.

2 MATERIAL AND METHODS

2.1 Performance of N fertilization on greenhouse experiment

2.1.1 Soil collection, characterization and base fertilization

The soil used at the greenhouse experiment is an Oxisol according to the Brazilian System of Soil Classification (DOS SANTOS, 2013). We chose a soil profile of ≈ 4 meters depth that has been digged under a native area within the Federal University of Lavras, MG, Brazil. Soil in the low portion (i.e. negligible organic matter content) of the mentioned profile was collected, air-dried and passed through a sieve of 2 mm. Granulometric and chemical analysis were performed according to Empresa Brasileira de Pesquisa Agropecuária - EMBRAPA (1999). Briefly, pH (5.9) was measured in a soil/water ratio of 1:2.5; organic matter (6.5 g kg^{-1}) by oxidation of O.M. with potassium dichromate; available P (0.84 mg kg^{-1}) and K (16 mg kg^{-1}) by Mehlich-1 extractor solution and quantified by flame photometry and colorimetry, respectively; exchangeable Ca^{2+} (0.53 $cmol_c$ kg^{-1}), Mg^{2+} (0.01 $cmol_c$ kg^{-1}) and Al^{3+} (0 $cmol_c$ kg^{-1}) by atomic absorption spectroscopy and titration for Al, using KCl 1 mol L^{-1} as extractor; H + Al (1.86 $cmol_c$ kg^{-1}) by NaOH titration of calcium acetate at buffered solution at pH 7. Effective CEC (0.64 $cmol_c$ kg^{-1}), CEC at pH 7.0 (2.51 $cmol_c$ kg^{-1}), sum of bases (0.58 $cmol_c$ kg^{-1}) and base saturation (23.15%) were indirectly calculated through potential acidity and exchangeable bases values. Granulometric analysis revealed 59% of clay, 24% of silt and 17% of sand by the pippete method.

Soil was weighted (4 kg pot^{-1}), limed to achieve base saturation of 70% using 1.26 g of calcium carbonate ($CaCO_3$) and 0.96 g of magnesium carbonate ($(MgCO_3)_4$ $Mg(OH)_2.5H_2O$), homogenized and incubated for 7 days. After that, a solution of 120 mg of Mg, 15 mg of Zn, 10 mg of Mn, 5 mg of Cu, 5 mg of B and

1 mg of Mo was applied per kg of soil, as magnesium sulfate ($MgSO_4$. $7H_2O$), zinc sulfate ($ZnSO_4.7H_2O$), manganese sulfate ($MnSO_4.H_2O$), coper sulfate ($CuSO_4.5H_2O$), boric acid (H_3BO_3) and sodium molybdate ($Na_2MoO_4.H_2O$). Then, soil was allowed to dry for few days following thoroughly mixture of K, P and Ca to the soil, as potassium phosphate (KH_2PO_4), calcium phosphate ($Ca(H_2PO_4)_2.H_2O$) and calcium sulfate ($CaSO_4$) respectively. Therefore, it was applied to the soil 300 mg kg^{-1} of K, 600 mg kg^{-1} of P, 774 mg kg^{-1} of Ca and 120 mg kg^{-1} of Mg in the liming and base fertilization steps, in addition to the micronutrients prior applied. All the reagents used at this experiment are of analytical grade and none of them have N on their compositions.

2.1.2 Experimental design

The experimental design consisted of a completely randomized factorial 14 x 4 with three repetitions. Treatments are the result of six fertilizers: granular urea, ammonium nitrate, polymer coated urea (multicote 4M®), polymer coated urea (urea + plastic resin), polymer coated urea (urea + polyurethane), urea formaldehyde, the mixing of CRF/SRF with granular urea (ratio of 40:60 in % of N and vice versa, totaling 8 treatments) therein called ‘blends’, and four rates of N (0, 150, 300 and 450 mg kg^{-1} of soil). Blends with Multicote 4M®, urea + plastic resin, urea + polyurethane and urea formaldehyde were represented by the following initial letters: M, A, B and C. The number followed by the letters means the percentage of N. Although urea + plastic resin is a fertilizer labeled to 9 months of release, it was included at this study since there is evidence that is would not reach this period labeled (CANCELLIER et al., in preparing). Urea + polyurethane is not a commercial product and was included at this study to verify its performance.

Treatments were applied after watering the pots intentionally to produce conditions susceptible for NH_3 volatilization. Irrigation was performed only two days after due to the low ambient temperature. Water was applied to reach 70% of field capacity.

2.1.3 Fertilizers characteristics

Granular urea: conventional N fertilizer containing 45% of N.
Ammonium nitrate: conventional N fertilizer containing 32% of N.
Multicote 4M®: controlled release fertilizer containing 40% of N and 2% of K_2O, produced by Haifa® and involved by polyurethane. Its release time according to the manufacturer is up to 4 months. The release of urea through a polyurethane membrane is affected by the amount of alkyl side chains as well as urethane content (WATANABE et al., 2009).
Urea + plastic resin: controlled release fertilizer containing 44% of N. The urea prill is involved by a plastic resin. Its release time according to the manufacturer is up to 9 months.
Urea + polyurethane: controlled release fertilizer containing 43.3% of N. This experimental product was considered at the present experiment as a potential fertilizer. Urea prills are involved by a resin of polyurethane.
Urea formaldehyde: slow release fertilizer containing 26% of N and 6% of K_2O. It's an urea-formaldehyde, which is formed by reaction of formaldehyde and urea (TRENKEL, 2010).

All controlled release fertilizers were submitted to scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS) analysis. The analysis were conducted in the 'Laboratório de microscopia eletrônica e análise ultra estrutural (LME)' in the Phytopathology department, Ufla. Fertilizers samples were cut with a scalpel, mounted on aluminum stubs and coated with carbon using a carbon evaporator (Union CED 020 model). Then, samples were observed through SEM (LEO EVO 40 XVP – Zeiss model), qualified and mapped regarding chemical composition by EDS (Quantax XFlash 5010 - Bruker equipment).

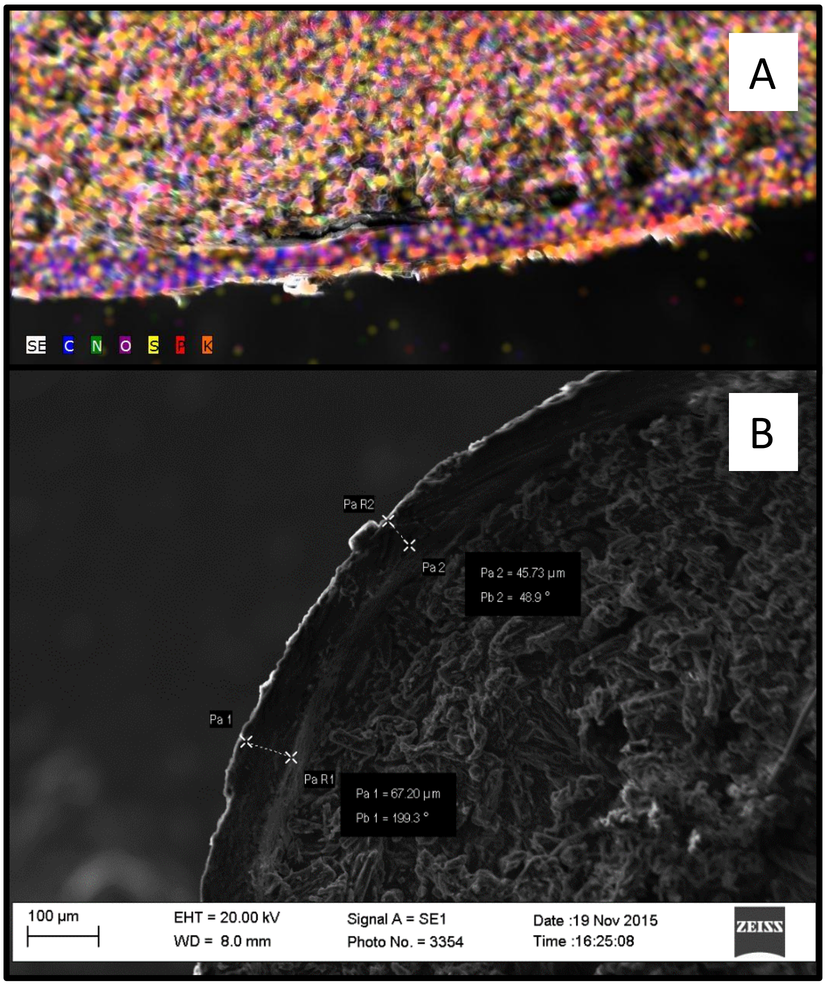

Figure 1 SEM images of multicote 4M®. EDS mapping of carbon, nitrogen, oxigen, sulfur, phosphorus and potash (A). Coated thickness between 45.73 and 67,20 µm (B)

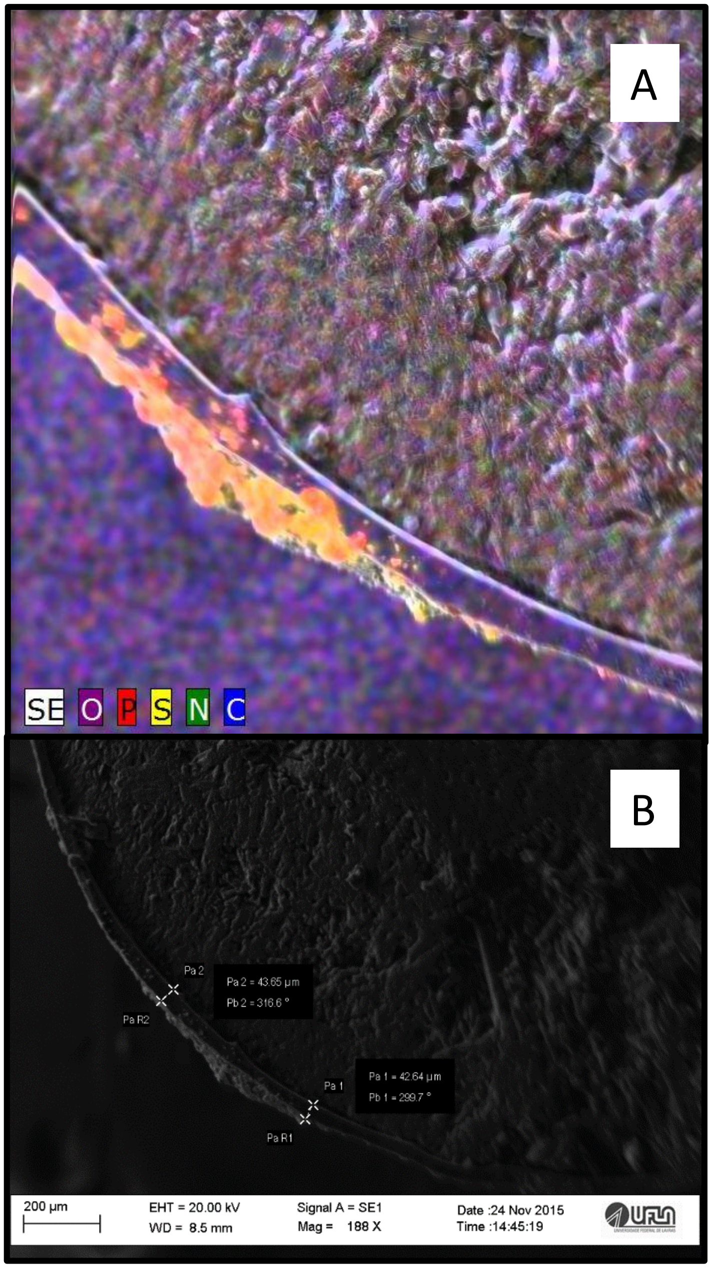

Figure 2 SEM images of urea + plastic resin. EDS mapping of oxygen, phosphorus, sulfur, nitrogen and carbon (A). Coated thickness between 43.65 and 42,64 µm (B)

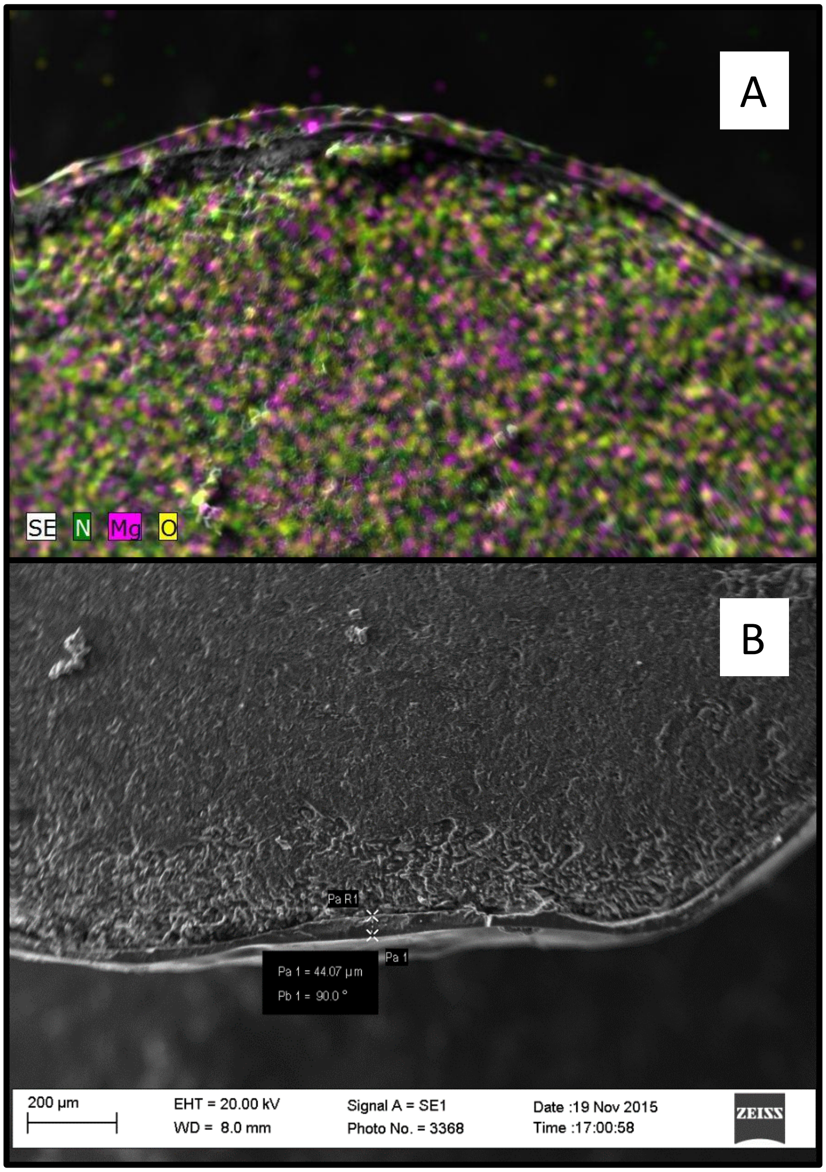

Figure 3 SEM images of urea + polyurethane. EDS mapping of nitrogen, magnesium and oxygen (A). Coated thickness equal to 44.07 µm (B)

2.1.4 Experimental conduction

Three successive cropping corn (Zea Mays, Dekalb, DKB 390 VT PRO2®, single cross hybrid) were carried out during a period of 35 days each in a fanned greenhouse. In all successive cropping, four seeds were sowed per pot, plants allowed to grow to ≈ 5 cm height, and thinned to two most vigorous plants per pot. The day of thinning was taken as the reference for the beginning of the cropping period and treatments were applied only in the 1st cropping, therefore, in the same day of the first thinning. In order to get a closely synchronism between the end of a successive crop (shoot harvesting operation) and the beginning of a successive crop (thinning operation), corn was sowed 7 days before shoots harvesting. Thus, we intended to avoid N release (susceptible to ammonia volatilization) by fertilizers with no plants for uptake. That did not occur since plants did not reach our prior criteria (plants about 5 cm height) for the beginning of a cropping, however a period of ``theoretically no uptake`` was shortened, since plants were ≈ 2 cm height. Shoot harvesting and sowing activities were carried out carefully to avoid any damage to fertilizers prills. To refrain possible damages by solar heating to PCF prills leaning in the edge of the pots, gentle beats were performed in the pots if fertilizers were dragged to the edge during watering. Watering was done so that all fertilizers prills were wetted.

Because two of the fertilizers used at the present experiment have K on their composition, a K leveling was conducted 11 days after fertilization as KCl in solution. Thus, all treatments were leveled to the same amount of K supplied at the maximum rate applied by urea formaldehyde. Thus, K was standardized to all treatments, and comparison among them allowed.

2.1.5 Plant analysis and agronomic parameters

Shoots were harvested, dried at 65°C in a forced air circulation dryer to constant weight, and weighted for dry mass. Subsequently, dried samples were ground in a Wiley mill and analyzed for N content according to Tedesco et al. (2005). Shoot N accumulation was calculated through the product of shoot dry mass (in g) and N content (mg g^{-1}).

Agronomic efficiency index (AEI) was calculated as follows:

$$AEI(\%) = \frac{[Shoots\ dry\ mass\ of\ N\!-\!fertilized\ treat.\left(\frac{g}{pot}\right) - Shoots\ dry\ mass\ of\ \ control\ (\frac{g}{pot})]}{[Shoots\ dry\ mass\ of\ N\!-\!urea\ treat.\left(\frac{g}{pot}\right) - Shoots\ dry\ mass\ of\ control\ (\frac{g}{pot})]} x100$$

The applied N recovery (ANR) was calculated as N accumulation of N-fertilized pots (g) minus N accumulation of control (g) divided by N rate (g).

SPAD reading were taken in the last expanded leaf using a chlorophyll meter atleaf®.

2.2 Nitrogen release curve in aqueous medium

2.2.1 Experimental apparatus

Ten grams of Multicote 4M®, urea + plastic resin and urea + polyurethane were placed into a bag and tied. Aqueous medium comprised two different pH, 5.5 and 6.5 (common range of pH adequate for cropping) adjusted with few drops of NaOH and HCl (≈0.01 mol L^{-1}) to distilled water; and two different ionic strengths, 0 (only distilled water) and 15 mM (equivalent to 0.8766 g L^{-1} of NaCl of analytical grade). 15 mM was chosen since it is the common ionic strength of weathered soils (CAMPOS et al., 2006), typical in Brazil. Thus, experimental design consisted of a completely randomized 4x3 scheme (4 aqueous medium and 3 PCUs) with 4 replications.

200 mL of aqueous medium were added into pots (previously weighted) and placed in a climatic camara set to 40 °C. Bags containing the PCUs were submerged into pots and N measuring were carried out over 231 days. Pots with new aqueous medium were acclimatized at least one day before the transference of bags to avoid temperature shock. N content was determined in the solution at room temperature through kjeldahl method following sulfuric digestion. For that, a sample was used for analysis and % of N release calculated based on the mass of the solution.

2.3 Statistical analysis

Data were submitted to normality (Shapiro-Wilk's test) and homogeneity (Barlett's test) tests. Then, data were submitted to analysis of variance and means were compared by Scott-Knott test at 5% of probability. To the greenhouse experiment, the effect of doses for each N source in each cropping was assessed by regression analysis. The best adjusted regression was defined among linear or quadratic models according to the greatest regression coefficient.

To N release test, means were compared by Scott-Knott test at 5% of probability for each fertilizer. The release behavior along time was assessed by regression chosen among linear, quadratic and exponential according with the greatest regression coefficient.

The analysis was performed using SISVAR 4.3 (Ferreira, 2011).

3 RESULTS

3.1 Nitrogen release test

Our nitrogen release test conducted in different pH and ionic strength of aqueous medium revealed that those parameters generally did not affect the N release (Table 1). N release of urea+polyurethane was affected in the days 1, 7 and 14 after incubation. Multicote 4M® in days 7 and 14 and urea + plastic resin only in the day 231 after incubation. Because different pH and ionic strength caused differences in N release in few days along the experiment, we considered all the four different aqueous medium plotted together to each fertilizer (Figure 1 and table 2).

Table 1 Nitrogen release experiment affected by pH (5.5 and 6.5) and ionic strength (0 and 15 mM) under 40° C

Fertilizer	pH/ Ionic Strength	Days after incubation														
		1	3	7	14	21	28	35	42	49	56	63	77	98	133	231
		N released (%)														
Urea+poly urethane	5.5/ 0mM	17.4 A	16.7 A	27.8 B	20.7 B	8.2 A	3.8 A	1.8 A	1.0 A	0.6 A	0.4 A	0.2 A	0.3 A	0.2 A	2.5 A	0.2 A
	5.5/ 15mM	14.5 B	16.8 A	29.7 A	21.8 A	8.7 A	4.1 A	1.8 A	1.1 A	0.6 A	0.4 A	0.2 A	0.2 A	0.2 A	1.5 B	0.1 A
	6.5/ 0mM	15.1 B	16.4 A	28.7 B	22.0 A	8.8 A	4.2 A	2.0 A	1.1 A	0.6 A	0.5 A	0.3 A	0.2 A	0.1 A	2.5 A	0.1 A
	6.5/ 15mM	14.9 B	16.8 A	29.8 A	21.4 A	8.7 A	4.1 A	2.0 A	1.1 A	0.6 A	0.4 A	0.2 A	0.2 A	0.1 A	1.5 B	0.04 A
Multicote 4M®	5.5/ 0mM	7.1 A	8.2 A	13.7 A	20.2 B	14.1 A	9.0 A	6.2 A	4.4 A	3.4 A	2.7 A	2.1 A	3.1 A	3.0 A	0.7 C	3.5 A
	5.5/ 15mM	7.4 A	9.3 A	14.1 A	22.2 A	13.4 A	8.8 A	5.9 A	4.0 A	3.2 A	2.6 A	2.0 A	2.8 A	2.9 A	0.5 C	3.1 A
	6.5/ 0mM	7.9 A	8.7 A	11.5 B	19.9 B	14.0 A	9.1 A	6.3 A	4.3 A	3.5 A	2.8 A	2.2 A	3.0 A	2.9 A	2.8 A	3.9 A
	6.5/ 15mM	6.9 A	9.0 A	13.0 A	20.6 B	14.7 A	9.0 A	6.4 A	4.8 A	3.5 A	2.7 A	2.1 A	2.7 A	2.8 A	1.6 B	3.3 A
Urea + plastic resin	5.5/ 0mM	3.1 A	4.8 A	15.6 A	33.1 A	13.6 A	7.4 A	4.6 A	3.7 A	2.5 A	1.9 A	1.4 A	2.0 A	1.8 A	0.1 A	1.4 B
	5.5/15mM	3.2 A	5.5 A	16.1 A	33.4 A	13.5 A	7.7 A	4.5 A	3.2 A	2.4 A	1.9 A	1.5 A	2.0 A	1.8 A	0.1 A	2.4 A
	6.5/ 0mM	3.0 A	5.7 A	16.6 A	33.7 A	12.8 A	6.5 A	4.7 A	3.2 A	2.4 A	1.9 A	1.4 A	2.0 A	1.7 A	0.2 A	1.4 B
	6.5/15mM	3.2 A	5.3 A	16.6 A	32.8 A	13.7 A	7.1 A	4.6 A	3.2 A	2.5 A	1.9 A	1.4 A	2.1 A	1.8 A	0.8 A	1.4 B

Letters compare fertilizers in each day after incubation according to Scott-Knott test ($p \leq 0{,}05$).CV=9.59.

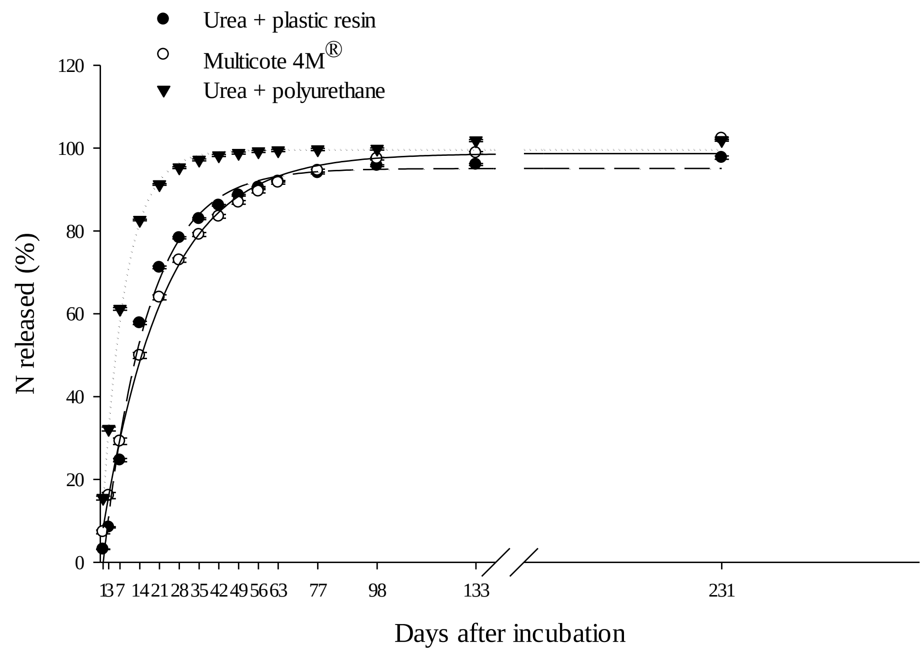

Figure 4 Cumulative nitrogen released by the fertilizers urea + plastic resin (----), multicote 4M® (__) and urea + polyurethane (….) in aqueous medium at 40° C

Table 2 Equations and R^2 for cumulative N release

	Equation	R^2	80% of N released (days)
Urea + plastic resin	% N released= 101.6117 * (1 - 0.9385^{days}) - 6.5388	0.99	30.065
Multicote 4M®	% N released = 94.6360 * (1 - 0.9558^{days}) + 4.0494	0.99	35.887
Urea+polyurethane	% N released = 95.3841 * (1 - 0.8849^{days}) + 4.2723	0.99	12.918

3.2 Nitrogen content in shoots

In the 1st cropping, granular urea and ammonium nitrate were similar ($P \leq 0.05$) and showed the highest values for N content in shoots compared to others fertilizers (Table 3). That was caused by the high solubility of conventional fertilizers. The fertilizers that produced the lowest values of N content in shoots were Multicote®, M60:U40 and Urea formaldehyde. For the 2nd cropping, Urea + plastic resin produced the highest value of N shoots content, by the other hand, granular urea, ammonium nitrate, M40:U60, B40:U60, B60:U40, Urea formaldehyde and its blends showed the lowest N shoots content values. Multicote®, Urea + plastic resin and urea + polyurethane were the fertilizers that showed the highest values of N in shoots, differently of granular urea, M60:U40, A40:U60, B40:U60, C40:U60 and C60:U40, which did not differ from each other and showed the lowest values in the 3rd cropping.

Table 3 Nitrogen content (mg g^{-1}) in corn shoots along three croppings as affected by different N sources

	Croppings		
	1st	2nd	3rd
	Shoot nitrogen content (mg g^{-1})		
Granular urea	22.05 Aa	10.28 Cb	7.52 Bc
Ammonium nitrate	22.78 Aa	11.25 Cb	8.12 Bc
Multicote 4M®	13.39 Fa	12.78 Ba	10.36 Ab
M 40: U 60	18.82 Ca	10.83 Cb	8.35 Bc
M 60: U 40	14.79 Fa	12.11 Bb	7.79 Bc
Urea + plastic resin	15.82 Ea	15.02 Aa	9.57 Ab
A 40: U 60	15.49 Ea	11.95 Bb	7.30 Bc
A 60: U 40	17.69 Ca	13.08 Bb	8.03 Bc
Urea + polyurethane	18.35 Ca	11.60 Bb	8.90 Ac
B 40: U 60	19.76 Ba	10.02 Cb	7.53 Bc
B 60: U 40	18.32 Ca	10.03 Cb	8.52 Bc
Urea formaldehyde	14.17 Fa	10.32 Cb	8.62 Bc
C 40: U 60	18.43 Ca	9.49 Cb	7.85 Bc
C 60: U 40	17.11 Da	9.44 Cb	7.69 Bc

Capital letters compare fertilizers in each cropping according to Scott-Knott test ($p \leq 0,05$). Lowercase letters compare croppings. M, A, B, C and U refer to multicote®, urea + plastic resin, urea + polyurethane, urea formaldehyde and granular urea, respectively. CV=12.26.

3.3 Shoots dry mass

In the first cropping, ammonium nitrate was the fertilizer that produced the maximum amount of dry mass, being Urea + plastic resin the source which yielded the lowest dry mass value (Table 4). Urea + plastic resin and Urea formaldehyde produced the maximum and minimum of dry mass, respectively, in the 2nd cropping. For the 3rd cropping, multicote® and urea + plastic resin caused the highest dry mass production and granular urea, ammonium nitrate, B40:U60, B60:U40, urea formaldehyde, C40:U60 and C60:U40 the lowest shoots dry mass.

For the cumulative dry mass, the sequence was ammonium nitrate = M60:U40 = A40:U60 = urea + polyurethane > granular urea = multicote 4M® = M40:U60 = urea + plastic resin = A60:U40 = B40:U60 = B60:U40 = C40:U60 > urea formaldehyde = C60:U40.

Table 4 Dry mass (g pot^{-1}) of corn shoots for each cropping and the sum of croppings as affected by different N sources

	Croppings			
	1st	2nd	3rd	Total
	Shoot dry mass (g pot^{-1})			
Granular urea	13.99 Ca	6.83 Cb	0.72 Dc	21.54 B
Ammonium nitrate	18.48 Aa	5.95 Db	1.32 Dc	25.75 A
Multicote 4M®	8.83 Fa	8.14 Ba	5.47 Ab	22.44 B
M 40: U 60	12.88 Da	6.64 Cb	2.20 Cb	21.72 B
M 60: U 40	13.56 Da	5.85 Db	3.87 Bb	23.29 A
Urea + plastic resin	5.75 Gb	10.41 Aa	5.50 Ab	21.66 B
A 40: U 60	14.67 Ca	7.22 Cb	2.73 Cc	24.62 A
A 60: U 40	11.66 Ea	7.48 Cb	3.65 Bc	22.79 B
Urea + polyurethane	12.31 Ea	9.21 Bb	2.20 Cc	23.72 A
B 40: U 60	12.54 Ea	7.41 Cb	0.97 Dc	20.92 B
B 60: U 40	13.07 Da	6.11 Db	1.51 Dc	20.69 B
Urea formaldehyde	15.09 Ca	0.94 Fb	1.05 Db	17.08 C
C 40: U 60	16.46 Ba	4.92 Db	1.01 Dc	22.39 B
C 60: U 40	13.36 Da	2.98 Eb	0.80 Dc	17.14 C

Capital letters compare fertilizers in each cropping according to Scott-Knott test ($p \leq 0,05$). Lowercase letters compare croppings. M, A, B, C and U refer to multicote 4M®, urea + plastic resin, urea + polyurethane, urea formaldehyde and granular urea, respectively. CV=20.20

In the 1st cropping, ammonium nitrate yielded the highest shoot dry mass in the rate of 292.83 mg kg^{-1} (27.23 g) (table 5), and the lowest one was produced by urea + plastic resin in the minor rate (3.89 g). Urea + plastic resin produced the highest shoot dry mass in the rate of 450 mg kg^{-1} (17.77 g), whereas urea formaldehyde produced only 0.42 g in the lowest rate in the 2nd cropping (table 6). In the 3rd cropping, the highest shoot dry mass was produced by fertilization of multicote 4M® at rate 450 mg kg^{-1} (10.66 g) and lowest shoot dry mass of 0.50 g was yielded by granular urea at rate of 300 mg kg^{-1} (table 7).

Among blends, A40:U60 produced the highest shoot dry mass (28.47 g) in the rate of 450 mg kg^{-1}, while A60:U40 the lowest one (10.69 g) in the 1st cropping (table 5). For 2nd cropping, the highest shoot dry mass was produced by A60:U40 at 450 mg kg^{-1} (15.93 g) and the lowest one by C40:U60 at 150 mg kg^{-1} (0.51 g) (table 6). 7.17 g was the highest shoot dry mass produced by M60:U40 at 450 mg kg^{-1}, and 0.70 g the lowest shoot dry mass when B40:U60 was used at 150 mg kg^{-1} for the 3rd cropping (table 7).

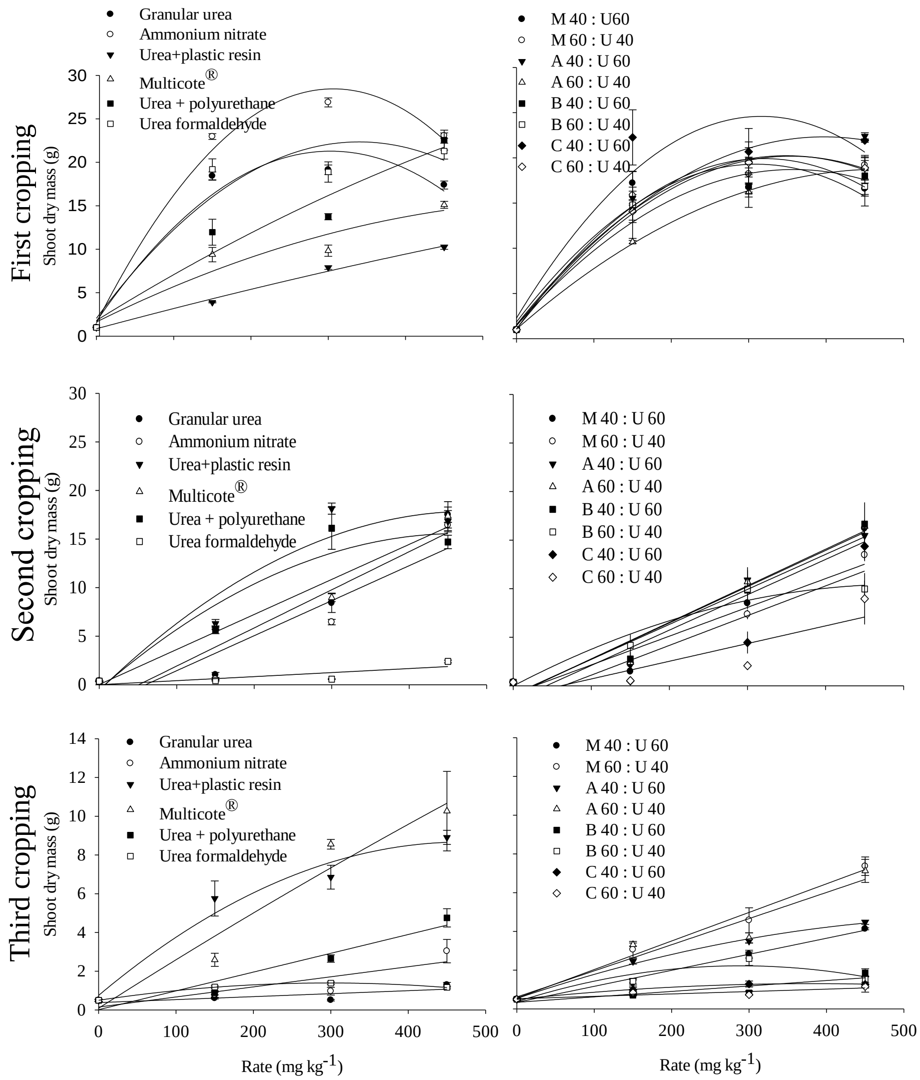

Figure 5 Dry mass along three corn croppings affected by N fertilizers (in the left) and their blends (in the right)

Table 5 Equations, R^2, adequate rate of N (ARN) and attainable dry mass (ADM) in the 1st cropping as affected by different N sources

	Dry mass in the 1st cropping			
	Equation	R^2	ARN (mg N kg^{-1})	ADM (mg)
Granular urea	$y = -0.0002N^2 + 0.1292N + 1.7025$	0.955	323.00	22.57
Ammonium nitrate	$y = -0.0003N^2 + 0.1757N + 1.5045$	0.987	292.83	27.23
Multicote 4M®	$y = -0.000034556N^2 + 0.0441N + 1.6285$	0.920	450	14.47
M 40: U 60	$y = -0.0002N^2 + 0.1127N + 1.8412$	0.923	281.75	17.72
M 60: U 40	$y = -0.0002N^2 + 0.1074N + 1.5438$	0.971	268.50	15.96
Urea + plastic resin	$y = -0.000006037N^2 + 0.0239N + 0.8525$	0.992	450	10.38
A 40: U 60	$y = -0.0001N^2 + 0.1051N + 1.4275$	0.986	450	28.47
A 60: U 40	$y = -0.000079222N^2 + 0.0749N + 1.0522$	0.999	450	18.71
Urea + polyurethane	$y = -0.000024N^2 + 0.0551N + 1.8007$	0.944	450	21.73
B 40: U 60	$y = -0.0001N^2 + 0.0965N + 1.442$	0.978	450	24.62
B 60: U 40	$y = -0.0002N^2 + 0.1181N + 1.0562$	0.999	295.25	18.49
Urea formaldehyde	$y = -0.0002N^2 + 0.1194N + 2.0507$	0.916	298.50	19.87
C 40: U 60	$y = -0.0002N^2 + 0.141N + 2.274$	0.897	352.50	27.13
C 60: U 40	$y = -0.0002N^2 + 0.1078N + 1.0745$	0.999	269.50	15.60

M, A, B, C and U in the first column refer to multicote 4M®, urea + plastic resin, urea + polyurethane, urea formaldehyde and granular urea, respectively.

Table 6 Equations, R^2, adequate rate of N (ARN) and attainable dry mass (ADM) in the 2nd cropping as affected by different N sources

	Dry mass in the 2nd cropping			
	Equation	R^2	ARN (mg N kg^{-1})	ADM (mg)
Granular urea	y = 0.0392N - 2	0.900	450.00	15.64
Ammonium nitrate	y= 0.036N - 2.1597	0.858	450.00	14.04
Multicote 4M®	y = 0.0361N + 0.021	0.966	450.00	16.27
M 40: U 60	y = 0.0364N - 1.543	0.924	450.00	14.84
M 60: U 40	y = 0.0296N - 0.8157	0.953	450.00	12.50
Urea + plastic resin	$y = -0.000080259N^2 + 0.0769N - 0.5865$	0.916	450	17.77
A 40: U 60	y = 0.036N - 0.8873	0.947	450.00	15.31
A 60: U 40	y = 0.0376N - 0.992	0.956	450.00	15.93
Urea + polyurethane	$y = -0.000074519N^2 + 0.0692N - 0.4847$	0.914	450	15.56
B 40: U 60	y = 0.0373N - 0.9843	0.962	450.00	15.80
B 60: U 40	$y = -0.000042111N^2 + 0.042N - 0.0225$	0.954	450	10.35
Urea formaldehyde	y = 0.0042N + 0.0047	0.682	450.00	1.89
C 40: U 60	y = 0.0306N - 1.9643	0.814	450.00	11.81
C 60: U 40	y = 0.0182N - 1.116	0.755	450.00	7.07

M, A, B, C and U in the first column refer to multicote 4M®, urea + plastic resin, urea + polyurethane, urea formaldehyde and granular urea, respectively.

Table 7 Equations, R^2, adequate rate of N (ARN) and attainable dry mass (ADM) in the 3rd cropping as affected by different N sources

	Dry mass in the 3rd cropping			
	Equation	R^2	ARN (mg N kg^{-1})	ADM (mg)
Granular urea	y = 0.0015N + 0.3795	0.602	450.00	1.05
Ammonium nitrate	y = 0.0052N + 0.1495	0.754	450.00	2.49
Multicote 4M®	$y = -4.2493(10^{-6})N^2 + 0.0254N + 0.0878$	0.949	450	10.66
M 40: U 60	y = 0.0083N + 0.337	0.988	450.00	4.07
M 60: U 40	y = 0.0147N + 0.5544	0.988	450.00	7.17
Urea + plastic resin	$y = -3.5607(10^{-5})N^2 + 0.0336N + 0.7496$	0.966	450	8.66
A 40: U 60	$y = -1.0829(10^{-5})N^2 + 0.0135N + 0.536$	0.996	450	4.42
A 60: U 40	y = 0.0135N + 0.6133	0.924	450.00	6.69
Urea + polyurethane	y = 0.0097N + 0.0134	0.930	450.00	4.38
B 40: U 60	y = 0.0028N + 0.3379	0.801	450.00	1.60
B 60: U 40	$y = -2.2096(10^{-5})N^2 + 0.0128N + 0.3708$	0.862	289.65	2.22
Urea formaldehyde	$y = -1.009(10^{-5})N^2 + 0.006N + 0.498$	0.999	297.32	1.39
C 40: U 60	$y = -5.5767(10^{-6})N^2 + 0.0043N + 0.4882$	0.998	385.53	1.32
C 60: U 40	y = 0.0012N + 0.5222	0.773	450.00	1.06

M, A, B, C and U in the first column refer to multicote 4M®, urea + plastic resin, urea + polyurethane, urea formaldehyde and granular urea, respectively.

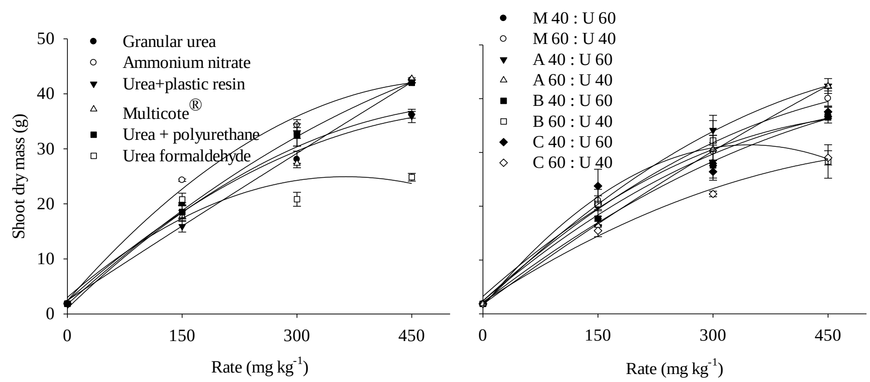

Figure 6 Total shoot dry mass affected by rates of N fertilizers (at left) and their blends (at right)

Table 8 Equations, R^2, adequate rate of N (ARN) and attainable dry mass (ADM) for total shoot dry mass accumulation along three croppings in corn as affected by different N sources

	Equation	R^2	ARN (mg N kg^{-1})	ADM (mg)
Granular urea	$y = -0.0001N^2 + 0,12N + 2.36$	0.99	450	38.09
Ammonium nitrate	$y = -0.0002N^2 + 0.16N + 2.38$	0.99	397.75	34.03
Urea + plastic resin	$y = -0.0001N^2 + 0.13N + 1.01$	0.98	450	41.24
Multicote 4M®	$y = -6.4344(10^{-6})N^2 + 0.09N + 2.46$	0.99	450	42.11
Urea + polyurethane	$y = -7.9496(10^{-5})N^2 + 0.1254N +1.7574$	0.99	450	42.09
Urea formaldehyde	$y = -0.0002N^2 + 0.12N + 2.99$	0.92	301.75	21.2
M40 : U60	$y = -0.0001N^2 + 0.12N + 2.40$	0.99	450	37.05
M60: U40	$y = -0.0001N^2 + 0.13N + 2.41$	0.99	450	40.34
A40: U60	$y = -0.0001N^2 + 0.14N + 1.77$	0.99	450	44.84
A60: U40	$y = -3.1281(10^{-5})N^2 + 0.1044N + 1.7522$	0.99	450	42.40
B40: U60	$y = -7.6839(10^{-5})N^2 + 0.1105N +2.1001$	0.99	450	36.26
B60: U40	$y = -0.0002N^2 +0.17N +1.40$	0.99	432.25	38.77
C40: U60	$y = -0.0001N^2 + 0.1271N + 3.2370$	0.94	450	40.18
C60:U40	$y = -7.6450(10^{-5})N^2 + 0.0932N + 2.1830$	0.99	450	28.64

M, A, B, C and U in the first column refer to multicote 4M®, urea + plastic resin, urea + polyurethane, urea formaldehyde and granular urea, respectively.

3.4 Nitrogen accumulation in shoots

Ammonium nitrate was the fertilizer that accumulated the greater amount of N in shoots in the 1st cropping and urea + plastic resin, the lowest (Table 9). In the 2nd cropping, urea + plastic resin showed the higher N accumulation and Urea formaldehyde and C60:U40 the lowest. Multicote 4M® and urea + plastic resin produced the highest N accumulation in the 3rd cropping, by the other hand the others sources produced the lowest values.

Cumulative N accumulation was in the order of ammonium nitrate > granular urea = urea + polyurethane = C40:U60 > M40:U60 = A40:U60 = A60:U40 = B40:U60 > Multicote 4M® = M60:U40 = urea + plastic resin = B60:U40 > C60:U40 > urea formaldehyde.

Table 9 Nitrogen accumulation (mg) in corn shoots for each cropping and the sum of croppings as affected by different N sources

	Croppings			Total
	1st	2nd	3rd	
	Shoot nitrogen accumulation (mg)			
Granular urea	366.85 Ba	79.06 Cb	5.71 Bc	451.62 B
Ammonium nitrate	509.44 Aa	96.53 Cb	11.58 Bc	617.56 A
Multicote 4M®	135. 41 Fa	124.66 Ba	66.11 Ab	326.18 D
M 40: U 60	284.77 Ca	80.73 Cb	19.84 Bc	385.34 C
M 60: U 40	232. 28 Ea	82.86 Cb	32.04 Bc	347.19 D
Urea + plastic resin	102.47 Gb	190.34 Aa	60.65 Ac	353.46 D
A 40: U 60	268.77 Da	89.07 Cb	21.23 Bc	379.08 C
A 60: U 40	246. 95 Ea	103.78 Cb	32.15 Bc	382.88 C
Urea + polyurethane	274.65 Da	125.97 Bb	22.04 Bc	422.67 B
B 40: U 60	298.42 Ca	86.75 Cb	7.58 Bc	392.75 C
B 60: U 40	267.20 Da	64.30 Cb	13.27 Bc	344.77 D
Urea formaldehyde	244.65 Ea	9.84 Eb	9.55 Bb	264.05 F
C 40: U 60	354.72 Ba	48.77 Db	8.25 Bc	411.74 B
C 60: U 40	270.76 Da	29.09 Eb	6.34 Bc	306.19 E

Capital letters compare fertilizers in each cropping according to Scott-Knott test ($p \leq 0,05$). Lowercase letters compare croppings. M, A, B, C and U refer to multicote 4M®, urea + plastic resin, urea + polyurethane, urea formaldehyde and granular urea, respectively. CV= 21.27

For the 1st cropping, ammonium nitrate caused the highest shoot N accumulation at rate of 379.91 mg kg^{-1} (797.67 mg), but Urea + plastic resin at rate of 150 mg kg^{-1} produced the lowest value, 75.59 mg (table 10). By the other hand, urea + plastic resin at rate of 450 mg kg^{-1} yielded the highest shoot N accumulation in the 2nd cropping (351.66 mg), and urea formaldehyde at 150 mg

kg^{-1}, the lowest one (4.95 mg) (table 11). In the 3rd cropping, the rate of 450 mg kg^{-1} produced the highest shoot N accumulation for fertilization with multicote 4M® (139.03 mg), and granular urea at rate of 300 mg kg^{-1}, the lowest (3.59 mg) (table 12). Among blends, C40:U60 yielded the highest shoot N accumulation at rate of 450 mg kg^{-1} (658.00 mg), and A60:U40 the lowest, 183.56 mg in the rate of 150 mg kg^{-1} for the 1st cropping (table 10). In contrast, A60:U40 yielded 225.15 mg at the highest rate, and C40:U60, the lowest (5.23 mg) at the minor rate for the 2nd cropping (table 11). In the 3rd cropping, A60:U40 again produced the highest shoot N accumulation at rate of 450 mg kg^{-1} (62.26 mg), and B40:U60 caused the lowest one (5.69 mg) at the minor rate (table 12).

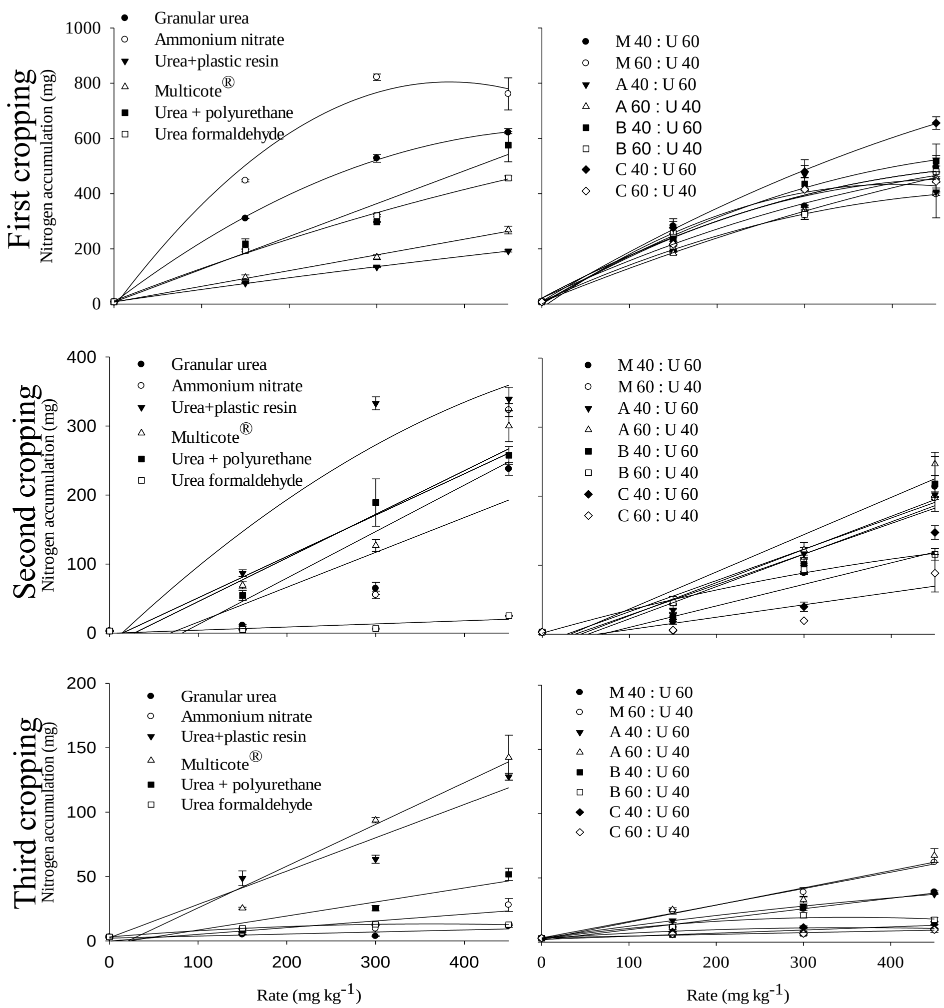

Figure 7 Nitrogen accumulation in corn shoots along three croppings affected by N fertilizers (in the left) and their blends (in the right)

Table 10 Equations, R^2, adequate rate of N (ARN) and attainable accumulation (AA) for N accumulation in the 1st cropping as affected by different N sources

	N accumulation in the 1st cropping			
	Equation	R^2	ARN (mg N kg^{-1})	AA (mg)
Granular urea	$y = -0.0023N^2 + 2.42N + 5.91$	0.99	450	627.54
Ammonium nitrate	$y = -0.0056N^2 + 4.25N - 10.59$	0.98	379.91	797.67
Multicote 4M®	$y = 0.5697N + 7.2205$	0.99	450.00	263.59
M 40: U 60	$y = -0.0015N^2 + 1.69N + 21.46$	0.97	450	477.94
M 60: U 40	$y = -0.0013N^2 + 1.45N + 9.17$	0.99	450	397.79
Urea + plastic resin	$y = -0.0001N^2 + 0.46N + 8.30$	0.99	450	193.52
A 40: U 60	$y = -0.0027N^2 + 2.22N - 13.82$	0.93	410.56	441.28
A 60: U 40	$y = -0.0006N^2 + 1.29N + 7.08$	0.99	450	465.05
Urea + polyurethane	$y = 1.1885N + 7.2401$	0.96	450.00	542.07
B 40: U 60	$y = -0.0016N^2 + 1.87N + 3.25$	0.99	450	519.85
B 60: U 40	$y = -0.0015N^2 + 1.6894N + 21.46$	0.97	450	477.94
Urea formaldehyde	$y = -0.0006N^2 + 1.23N + 11.35$	0.99	450	443.75
C 40: U 60	$y = -0.0010N^2 + 1.89N + 9.78$	0.99	450	658.00
C 60: U 40	$y = -0.0020N^2 + 1.90N - 0.14$	0.98	450	450.98

M, A, B, C and U in the first column refer to multicote 4M®, urea + plastic resin, urea + polyurethane, urea formaldehyde and granular urea, respectively.

Table 11 Equations, R^2, adequate rate of N (ARN) and attainable accumulation (AA) for N accumulation in the 2nd cropping as affected by different N sources

	N accumulation in the 2nd cropping			
	Equation	R^2	ARN (mg N kg^{-1})	AA (mg)
Granular urea	y= 0.5053N -34.6450	0.802	450.00	192.74
Ammonium nitrate	y= 0.6737N -55.0612	0.728	450.00	248.10
Multicote 4M®	y= 0.6325N -17.6527	0.925	450.00	266.97
M 40: U 60	y= 0.4685N -24.6723	0.890	450.00	186.15
M 60: U 40	y= 0.4429N -16.7805	0.944	450.00	182.52
Urea + plastic resin	$y = -0.0009N^2 + 1.2248N - 17.252$	0.908	450	351.66
A 40: U 60	y= 0.4526N -12.7636	0.965	450.00	190.91
A 60: U 40	y= 0.5394N -17.5846	0.949	450.00	225.15
Urea + polyurethane	y= 0.5990N -8.8080	0.971	450.00	260.74
B 40: U 60	y= 0.4803N -21.3225	0.921	450.00	194.81
B 60: U 40	$y = -0.0002N^2 + 0.3582N + 1.35$	0.993	450	122.04
Urea formaldehyde	y= 0.0454N -0.3693	0.735	450.00	20.06
C 40: U 60	y= 0.3118N -21.3771	0.793	450.00	118.93
C 60: U 40	y= 0.1798N -11.3598	0.753	450.00	69.55

M, A, B, C and U in the first column refer to multicote 4M®, urea + plastic resin, urea + polyurethane, urea formaldehyde and granular urea, respectively.

Table 12 Equations, R^2, adequate rate of N (ARN) and attainable accumulation (AA) for N accumulation in the 3rd cropping as affected by different N sources

	N accumulation in the 3rd cropping			
	Equation	R^2	AAR (mg N kg^{-1})	AA (mg)
Granular urea	$y= 0.0159N + 2.1311$	0.63	450.00	9.29
Ammonium nitrate	$y= 0.0520N -0.1098$	0.81	450.00	23.29
Multicote 4M®	$y= 0.3241N -6.8181$	0.97	450.00	139.03
M 40: U 60	$y= 0.0815N + 1.5008$	0.99	450.00	38.18
M 60: U 40	$y= 0.1278N + 3.2796$	0.99	450.00	60.79
Urea + plastic resin	$y= 0.2588N + 2.4281$	0.95	450.00	118.89
A 40: U 60	$y = -0.00004N^2 + 0.0982N + 3.05$	1.00	450.00	37.17
A 60: U 40	$y= 0.1338N + 2.0497$	0.94	450.00	62.26
Urea + polyurethane	$y= 0.1089N -2.4587$	0.92	450.00	46.55
B 40: U 60	$y= 0.0244N + 2.0859$	0.85	450.00	13.07
B 60: U 40	$y = -0.0001N^2 + 0.0962N + 2.41$	0.96	450.00	25.45
Urea formaldehyde	$y = -0.00007N^2 + 0.0555N + 3.07$	0.99	363.06	13.15
C 40: U 60	$y = -0.00006N^2 + 0.0473N + 2.87$	0.99	354.71	11.26
C 60: U 40	$y = -0.000001N^2 + 0.0138N + 3.31$	0.93	450	9.30

M, A, B, C and U in the first column refer to multicote 4M®, urea + plastic resin, urea + polyurethane, urea formaldehyde and granular urea, respectively.

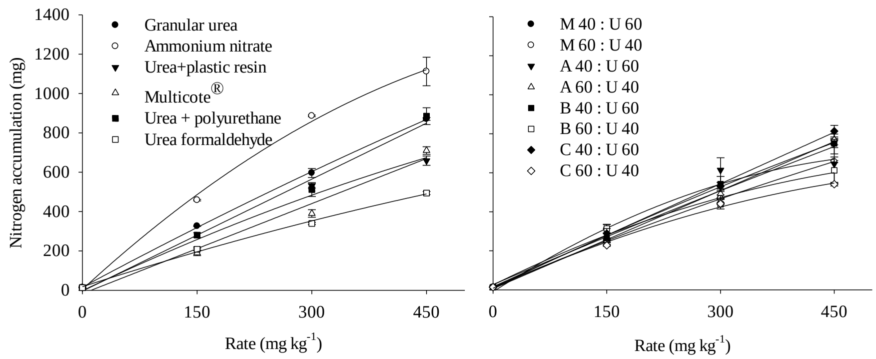

Figure 8 Total nitrogen accumulation for N fertilizers (at left) and their blends (at right)

Table 13 Equations, R^2, adequate rate of N (ARN) and attainable N accumulation (ANA) for total shoot dry mass accumulation along three croppings in corn as affected by different N sources

	Equation	R^2	ARN (mg N kg^{-1})	ANA (mg)
Granular urea	$y = -0.0004N^2 + 2.0827N + 16.1470$	0.999	450	872.36
Ammonium nitrate	$y = -0,0024N^2 + 3.5753N + 4.5071$	0.997	450	1127.39
Urea + plastic resin	$y = -0.0008N^2 + 1.8477N – 1.8982$	0.981	450	667.57
Multicote 4M®	$y = 1.5263N – 17.2502$	0.979	450	669.58
Urea + polyurethane	$y = 1.8964N – 4.0265$	0.989	450	849.35
Urea formaldehyde	$y = -0.0004N^2 + 1.2492N + 18.1796$	0.996	450	499.32
M 40 : U 60	$y = -0.0002N^2 + 1.6655N + 26.9835$	0.987	450	735.96
M 60: U 40	$y = -0.0005N^2 + 1.6639N + 13.4456$	1.00	450	660.95
A 40: U 60	$y = -0.0022 N^2 + 2.5019N – 10.2318$	0.958	450	670.12
A 60: U 40	$y = 1.6759N + 5.7935$	0.998	450	759.95
B 40: U 60	$y = -0.0005N^2 + 1.8742N + 8.9399$	0.998	450	751.08
B 60: U 40	$y = -0.0014N^2 + 1.9152N + 25.0784$	0.986	450	603.42
C 40: U 60	$y = 1.7600N + 15.7523$	0.999	450	807.75
C 60:U 40	$y = -0.0013N^2 + 1.7665N + 8.2886$	0.996	450	539.96

M, A, B, C and U refer to multicote 4M®, urea + plastic resin, urea + polyurethane, urea formaldehyde and granular urea, respectively.

3.5 SPAD index

In the 1st cropping, ammonium nitrate and granular urea showed the greatest values for SPAD reading, and urea + plastic resin the lowest value (Table 14). By the other hand, urea + plastic resin was higher in SPAD values, and urea formaldehyde the lower SPAD reading among treatments for the 2nd cropping. In the 3rd cropping, multicote 4M®, M60:U40, urea + plastic resin and A60:U40 were greatest in SPAD readings but granular urea and ammonium nitrate showed the lowest values.

Table 14 Chorophyll content (SPAD) along three croppings in corn shoots as affected by different N sources

	Croppings		
	1st	2nd	3rd
	Chorophyll content (SPAD)		
Granular urea	29.56 Aa	9.59 Eb	2.56 Ec
Ammonium nitrate	30.89 Aa	10.70 Eb	3.04 Ec
Multicote 4M®	20.49 Ea	17.21 Cb	13.42 Ac
M 40: U 60	28.10 Ba	13.80 Db	7.24 Cc
M 60: U 40	25.98 Ca	15.06 Db	12.08 Ac
Urea + plastic resin	17.28 Fb	21.33 Aa	13.03 Ac
A 40: U 60	26.76 Ca	15.63 Cb	9.56 Bc
A 60: U 40	25.06 Da	18.29 Bb	11.92 Ac
urea + polyurethane	24.35 Da	15.85 Cb	9.27 Bc
B 40: U 60	26.53 Ca	14.22 Db	6.19 Cc
B 60: U 40	24.15 Da	15.46 Cb	5.74 Cc
Urea formaldehyde	23.83 Da	3.78 Gb	3.78 Db
C 40: U 60	24.52 Da	8.47 Fb	4.74 Dc
C 60: U 40	27.73 Ba	7.91 Fb	4.54 Dc

Capital letters compare fertilizers in each cropping according to Scott-Knott test ($p \leq 0,05$). Lowercase letters compare croppings. M, A, B, C and U refer to multicote 4M®, urea + plastic resin, urea + polyurethane, urea formaldehyde and granular urea, respectively. CV= 12.25.

3.6 Agronomic efficiency index (AEI)

For the 1st cropping, all fertilizers were similar regarding AEI, except multicote 4M® and urea + plastic resin (Table 15). Multicote 4M®, urea + plastic resin, A40:U60, A60:U40, urea + polyurethane, B40:U60, B60:U40 had the highest values for the 2nd cropping and urea formaldehyde, the lowest. In the 3rd cropping, multicote 4M®, M60:U40, urea + plastic resin, A40:U60, A60:U40 showed the highest values but granular urea revealed the lowest AEI.

Table 15 Agronomic efficiency index (AEI) along three croppings in corn shoots as affected by different N sources

	Croppings			
	1st	2nd	3rd	Total
	AEI (%)			
Granular urea	100.00 Aa	100.00 Ba	100.00 Ea	100.00 C
Ammonium nitrate	134.74 Aa	71.90 Cb	475.35 Da	122.26 A
Multicote 4M®	60.97 Bc	458.69 Ab	3704.76 Aa	101.56 C
M 40: U 60	91.63 Ab	137.61 Bb	1233.88 Ba	100.87 C
M 60: U 40	97.27 Ab	209.50 Bb	2588.99 Aa	108.75 B
Urea + plastic resin	37.07 Cc	548.47 Ab	3577.88 Aa	98.57 C
A 40: U 60	105.88 Ab	195.17 Ab	1916.79 Aa	114.29 B
A 60: U 40	82.71 Ac	222.41 Ab	2393.04 Aa	102.38 C
urea + polyurethane	88.48 Ac	497.24 Ab	973.29 Ba	108.68 B
B 40: U 60	89.24 Ab	283.74 Aa	250.10 Da	95.47 C
B 60: U 40	92.57 Ac	272.20 Ab	1249.17 Ba	98.13 C
Urea formaldehyde	109.01 Ab	7.74 Ec	550.59 Ca	81.30 D
C 40: U 60	119.74 Ab	61.21 Dc	435.81 Ca	106.33 C
C 60: U 40	95.47 Ab	37.81 Dc	233.17 Da	77.35 D

Capital letters compare fertilizers in each cropping according to Scott-Knott test ($p \leq 0,05$). Lowercase letters compare croppings. M, A, B, C and U refer to multicote 4M®, urea + plastic resin, urea + polyurethane, urea formaldehyde and granular urea, respectively. CV= 11.30. For total IEA, CV=11.59.

3.7 Applied nitrogen recovery (ANR)

Ammonium nitrate proportionated the maximum ANR and urea + plastic resin the least ANR in the 1st cropping (Table 16). By the other hand, urea + plastic resin had the greatest ANR value but urea formaldehyde and C60:U40, the lowest in the 2nd cropping. In the 3rd cropping, multicote 4M® and urea + plastic resin showed the greatest values and granular urea, ammonium nitrate, B40:U60, B60:U40, urea formaldehyde and its blends the lowest.

Table 16 Accumulated nitrogen recovery (ANR) along three croppings in corn shoots as affected by different N sources

	Croppings			
	1st	2nd	3rd	Total
	ANR (%)			
Granular urea	42.60 Ba	6.52 Cb	0.29 Cc	49.39 B
Ammonium nitrate	60.96 Aa	7.50 Cb	0.80 Cc	69.26 A
Multicote 4M®	14.20 Ga	12.62 Ba	6.34 Ab	33.16 F
M 40: U 60	33.88 Da	7.12 Cb	1.77 Bc	42.76 D
M 60: U 40	26.68 Fa	7.71 Cb	3.27 Bc	37.65 E
Urea + plastic resin	10.68 Hb	20.04 Aa	6.51 Ac	37.24 E
A 40: U 60	30.41 Ea	8.62 Cb	2.09 Bc	41.12 D
A 60: U 40	27.28 Fa	10.09 Cb	3.25 Bc	40.63 D
urea + polyurethane	30.21 Ea	12.75 Bb	1.80 Bc	44.76 C
B 40: U 60	33.84 Da	7.95 Cb	0.47 Cc	42.26 D
B 60: U 40	31.37 Ea	6.96 Cb	1.25 Cc	39.58 D
Urea formaldehyde	27.32 Fa	0.63 Eb	0.82 Cb	28.77 G
C 40: U 60	39.99 Ca	3.83 Db	0.65 Cc	44.46 C
C 60: U 40	30.96 Ea	2.20 Eb	0.39 Cb	33.56 F

Capital letters compare fertilizers in each cropping according to Scott-Knott test ($p \leq 0,05$). Lowercase letters compare croppings. M, A, B, C and U refer to multicote 4M®, urea + plastic resin, urea + polyurethane, urea formaldehyde and granular urea, respectively. CV=16.89. For total accumulated nitrogen recovery, CV=9.26.

4 DISCUSSION

4.1 Effect of N fertilization along three corn croppings

As expected, granular urea and ammonium nitrate were ranked among treatments with the highest values in the 1st cropping for most of the parameters studied. Those fertilizers caused the highest N content and SPAD readings among the others fertilizers, being ammonium nitrate numerically higher than granular urea. For AEI, conventional fertilizers were similar to most fertilizers, being ammonium nitrate ≈ 35% higher than granular urea and 263% higher than urea + plastic resin, which showed the lowest AEI. Because ammonium nitrate was numerically or statistically higher than granular urea in the 1st cropping, that suggests that our experimental condition was propitious for NH_3 losses, as losses by denitrification do not occur in aerobic conditions and leaching is not considered for our pot experiment. Thus, differences among treatments are likely to be caused by different NH_3 volatilization rates along three croppings of the present experiment, in addition to different N release rates intrinsically associated to sources.

The variation of N content, shoot dry mass, N accumulation, SPAD readings, AEI and ANR in the 1st cropping among treatments was in the range of 70, 221, 397, 178, 263 and 470% which show a wide range of N availability of treatments for the 1st cropping. In the 3rd cropping the variation was 42, 664, 1057, 424, 3604 and 2145%. The larger variation in the 3rd cropping compared to the 1st one for all variables, except N content, means that N sources effectively were able to extend the release of the nutrient up to the end of the experiment. The reasons for a wide variation in the 1st and 3rd cropping are: wide range of N release capacity of the fertilizers, soil with low organic matter content that could diminish differences due to mineralization, and the absence of leaching which could enlarge the differences at least for the 1st cropping for granular urea and ammonium nitrate mainly.

Because dry mass and N accumulation decreased after the rate of 300 mg kg^{-1} for the 1st cropping for ammonium nitrate, that means that the critical level was achieved and we satisfactory comprised the range of N rates in corn for the experimental condition.

Under field conditions, several works have not showed differences among different N sources, contrary to the present experiment. For example, Silva et al. (2012a) did not find differences regarding foliar N content, N accumulated in dry mass, dry mass and green mass. Pereira et al. (2009) did not find differences in leaf N content among fertilized plots with conventional urea, stabilized urea and polymer-coated urea at different rates. Frazão et al. (2014) did not find differences in shoot dry mass and foliar N content testing stabilized urea, conventional and PCU. Valderrama et al. (2014) did not find differences in foliar chlorophyll index and foliar N content among conventional and polymer-coated urea in two growing seasons of corn. Martins, Cazetta and fukuda (2014) did not find differences in foliar N content and shoot dry mass when polymer and conventional urea were applied under a rainy period but found differences among sources for shoot dry mass when fertilizers were applied under a dryer condition after fertilization.

Urea formaldehyde caused similar shoot dry mass compared to granular urea and C40:U60 was even higher than granular urea in the 1st cropping. Shoot dry mass and N accumulation for urea formaldehyde and its blends was lower than granular urea in the 2nd cropping. This may be ascribed to the higher NH_3 volatilization of urea formaldehyde. Viero et al. (2015) concluded that the slow release urea did not reduce NH_3 volatilization in the field compared to conventional urea.

In contrast to the 1st cropping, granular urea and ammonium nitrate did not show the highest values for all variables among others treatments in the 2nd cropping. By the other hand, urea + plastic resin had the highest N content, dry mass, N accumulation, SPAD reading and ANR in the 2nd cropping, revealing that urea + plastic resin provided more N in the 2nd cropping compared to the others sources. That was caused by the longer N release claimed by the manufacturer (9 months) and shows that urea + plastic resin released most of N in the 2nd cropping. For AEI, urea + plastic resin was similar to its blends, multicote 4M®, urea + polyurethane, B40:U60 and B60:U40, which means similar N nutrition capacity by different sources and blends, revealing possibility of decreasing costs by mixing CRF with granular urea or by using different N fertilizers.

For the 3rd cropping, multicote 4M® and urea + plastic resin had the highest dry mass, N accumulation and ANR, which shows that those fertilizers provided the highest N availability among the others sources. For N content, urea + polyurethane also demonstrated similar results to multicote 4M® and urea + plastic resin, being those the highest among others sources. SPAD readings posed multicote 4M®, M60:U40, urea + plastic resin and A60:U40 with the highest values among the others sources. In respect to AEI, multicote 4M®, M60:U40, urea + plastic resin and its blends were similar. That suggests that different controlled release fertilizers are effective in releasing N along the studied period, and blends are efficient as well, depending on the variable analyzed.

When all three croppings are considered for calculating total N accumulation, AEI and ANR, ammonium nitrate was the best source. It is likely that under field condition the controlled release fertilizers would surpass this conventional fertilizer, as fertilizers leaching did not occur in this experiment, likely enhancing the N accumulation, AEI and ANR index of ammonium nitrate. When shoot dry mass is accounted for the entire experiment, M60:U40, A40:U60 and urea + polyurethane are similar to ammonium nitrate, which means there is the possibility of obtaining similar dry mass amount by using controlled release fertilizers or blends.

4.2 Incubation of fertilizers in aqueous medium

Our release test revealed that multicote 4M® and urea + plastic resin were more effective in controlling the N release than urea + polyurethane. This is consistent with higher N accumulation, shoot dry mass and ANR of multicote 4M® and urea + plastic resin than urea + polyurethane. Higher SPAD readings of multicote 4M®, M60:U40, urea + plastic resin and A60:U40 in addition to the higher AEI of urea + plastic resin and its blends, multicote 4M® and M60:U40 than urea + polyurethane or its blends in the 3rd cropping suggest a greater effect of N fertilization by the mentioned sources than urea + polyurethane. However, urea + polyurethane showed similar N content compared to urea + plastic resin and multicote 4M® in the 3rd cropping, which may be related to a dilution effect when plants produce high ow low biomass.

4.3 Water as a driving factor for non-responsive N fertilizers

Water is essential for dissolution and incorporation of fertilizers, nutrient uptake and crop growth, since it is the medium that nutrients are dissolved in. At this view, water contributes for increasing N availability when 1) decreases NH_3 volatilization when urea is incorporated into soil by rainfall or irrigation, 2) decreases N leaching when rainfall is not enough for this and 3) determining soil humidity. Thus, the use of N is related to rainfall or irrigation during the experiment conduction. A greenhouse experiment is likely to represent a harsher condition compared to field trial since it depends of daily watering. Daily irrigations are necessary since plants are affected by soil water content due to low soil volume for roots. Rampim et al. (2010) did not find differences regarding N content and dry mass when urea, urea with urease inhibitor, mix of ammonium sulfate and urea, ammonium sulfate and urea mixed with oil was applied in a sandy soil with 13.67 g dm-3 of SOM content in a greenhouse experiment. According to the authors, frequent pot irrigations cause no differences to corn crop regarding N use. Silva et al. (2012b) in a soil with 63% of sand and 21 mg dm-3 of SOM content did not find differences in N content in dry mass when monoammonium phosphate (MAP) and two polymered MAP were applied.

Soil humidity or rainfall occurrence have been found to influence N use by plants. For example, in the experiment of Frazão et al. (2014), it was found higher corn yield when urea with urease inhibitor was used compared to polymer-coated urea, and the authors ascribed this result to the lack of rainfall during the first three days after fertilization, high temperatures and soil humidity. Viero et al. (2015) found a reduction of volatilization peaks when 10 mm of irrigation was applied after fertilization with conventional urea, slow release urea and urea with urease inhibitor compared to irrigation prior fertilization and without irrigation. Cumulative losses from conventional urea was reduced by irrigation after fertilization by 65%, for urea with urease inhibitor, 60% and 50% for slow release urea, according to the authors. Noellsch et al. (2009) found higher corn yield of mix of polymer-coated urea with conventional urea treatment (50/50 w/w) compared to conventional urea only in 2006 but that did not occur in 2005. The authors suggested this to the greater rainfall in 2006 that influence the release of different fertilizers. In the dryer year of 2005, Noellsch et al. (2009) found higher N uptake for PCU in a wetter area of the experiment, and suggested this to lower losses compared to urea. Valderrama et al. (2014) did not find differences in chlorophyll index, foliar N content and grain yield in two growing season in corn when applied three polymer coated urea and conventional urea before irrigation. The authors suggest that coating was not efficient due to the higher temperatures, however, the availability of irrigation done when necessary was likely the cause of no differences among sources. Martins, Cazetta and Fukuda (2014) found benefits in using polymer-coated urea followed by insignificant rainfall during 15 days after application, but when conventional urea and PCU were split under considerable rainfall occurrence, there were no differences among sources, and the authors suggest that conventional urea and PCU are more inclined to similar results under irrigation or rainy season.

At the present experiment it was found high differences among fertilizers, which in the first view, it is due to the wide N release capacity of the fertilizers used. Because daily watering is thought to dissolve the polymer-coated urea in a rate greater than under field conditions, we speculate that irrigation or rainfall occurrence are not the only factor responsible for no differences among different N fertilizers in the experiments reported by the literature. Among many factors playing a role on crops, SOM should be a concern.

4.4 SOM content as a possible factor for non-responsive N fertilizers

SOM contributes to N nutrition of plants as N-organic compounds are mineralized to mineral forms of NH_4^+ and NO_3^-. Therefore, SOM should be accounted for N management as well as any others N-credits when supplied to plants. Thus, there is the possibility of reducing the use of mineral fertilizers avoiding fertilization in excess and environmental pollution risks.

Predicting the contribution of SOM to N nutrition of plants prior N fertilization is a hard task since that is a phenomenon tightly related to biological factors as well as climate conditions. Because of that, few works have considered the SOM content on their discussions (ZAVASCHI et al., 2014; MOTA et al., 2015) in an attempt to explain the lack of differences among different N treatments in respect to crops response. Beyond the mineralization of SOM *per se*, Zavaschi et al. (2014) include the priming effect of N fertilization upon mineralization, justified by no statistical differences found in corn yield among control and N treatments with conventional urea and polymer-coated urea in different rates in a no-till system during 18 years with 28 g dm-3 of SOM content in the upper layer. Qiu et al. (2016) found during a 250 days of incubation experiment cumulative CO_2 emissions (mineralization) of 9% greater when N was added to soil compared to control, higher emissions when N was added to soil with maize residues after 20 days of incubation compared to soil with maize residues only and 6% more microbial biomass carbon when soil was treated with N compared to control. Because the soil of our experiment was collected in deeper layer in the view of a negligible SOM content (later revealed by the soil analysis as 6.5 g kg^{-1}), we speculate that differences among fertilized treatments in the 3rd cropping in respect to N content, dry mass, N accumulation, SPAD reading, AEI and ANR in the range of 42%, 664%, 1.057%, 424%, 3.600% and 2.140% respectively are not only caused by different N fertilizers, but also part of the low contribution of SOM to crop nutrition. Rampim et al. (2010) did not find differences regarding N content and dry mass when urea, urea with urease inhibitor, mix of ammonium sulfate and urea, ammonium sulfate and urea mixed with oil was applied in a sandy soil with 13.67 g dm-3 of SOM content in a greenhouse experiment. Silva et al. (2012b) in a soil with 63% of sand and 21 mg dm-3 of SOM content did not find differences in N content in dry mass when monoammonium phosphate (MAP) and two polymered MAP were applied. By the other hand, accumulation of N in dry mass was affected by rates of the fertilizers, according to the authors.

5 CONCLUSIONS

The greatest values of N content, shoot dry mass, N accumulation, SPAD index and ANR were caused by granular urea and ammonium nitrate in the 1^{st} cropping, by the other side, controlled release fertilizers and/or theirs blends caused the highest values for the mentioned variables in the 2^{nd} and 3^{rd} croppings.

Some blends caused similar SPAD index values in the 3^{rd} cropping and AEI in the 2^{nd} and 3^{rd} croppings compared to controlled release fertilizers applied solely, which suggest possibility of reducing costs of fertilizers.

The range of pH and ionic strength used at the release test did not affect the N release from controlled release fertilizers for most of the days. The N release curve followed an exponential model.

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