



Use of Ammonium Sulfate in Soil Produced with Residual Ammonia Liquor from the Steel Industry and Residual Sulfuric Acid from Automotive Batteries in the Initial Development of Maize (*Zea Mays L.*)

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Utilização de sulfato de amônio no solo produzido com licor de amônia residual da indústria de produção de aço e ácido sulfúrico residual de baterias automotivas no desenvolvimento inicial da cultura do milho (*Zea mays L.*)

RESUMO

Objetivo – Este trabalho tem por objetivo avaliar a viabilidade técnica da produção do sulfato de amônio a partir de resíduos industriais e analisar o seu desempenho agrônomo como fertilizante de nitrogênio e enxofre na cultura de milho.

Metodologia – A pesquisa adotou uma abordagem experimental baseada em ensaios controlados em vasos. O sulfato de amônio foi produzido utilizando ácido sulfúrico residual proveniente de baterias automotivas e licor amoniaco da indústria siderúrgica. Uma segunda formulação foi preparada empregando ácido residual e amônia comercial, enquanto o sulfato de amônio (P.A.) foi utilizado como referência. As soluções de fertilizantes foram aplicadas ao solo em doses de nitrogênio variando de 20 a 120 kg N·ha⁻¹, seguidas da semeadura do milho (*Zea mays L.*). O crescimento das plantas e o acúmulo de biomassa foram posteriormente avaliados.

Originalidade/Relevância – O estudo aborda uma lacuna relevante relacionada ao reaproveitamento de resíduos industriais para a produção de fertilizantes, contribuindo para as discussões sobre a redução da dependência do Brasil em relação aos fertilizantes importados. O estudo aprofunda o conhecimento sobre fontes alternativas de nutrientes alinhado aos princípios da economia circular.

Resultados – As plantas de milho apresentaram respostas positivas a todas as fontes de sulfato de amônio testadas. No entanto, taxas de aplicação mais elevadas resultaram em efeitos fitotóxicos, especialmente na biomassa aérea, enquanto a biomassa radicular foi negativamente afetada mesmo em doses mais baixas de nitrogênio.

Contribuições Teóricas/Metodológicas – Os resultados contribuem para a compreensão da dinâmica de nutrientes e dos limites de toxicidade associados a fontes alternativas de sulfato de amônio, oferecendo subsídios metodológicos para a avaliação de fertilizantes derivados de resíduos em condições controladas.

Contribuições Sociais e Ambientais – A abordagem proposta promove a valorização de resíduos industriais, potencialmente reduzindo passivos ambientais e contribuindo para cadeias de suprimento de fertilizantes mais sustentáveis. Além disso, apoia estratégias voltadas ao fortalecimento da sustentabilidade agrícola e ao fomento de um sistema fornecimento de insumos agrícolas mais resiliente e autônomo.

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Palavras-chave: Resíduos industriais, fertilidade do solo, desenvolvimento das culturas, fertilizantes alternativos.

Use of Ammonium Sulfate in Soil Produced with Residual Ammonia Liquor from the Steel Industry and Residual Sulfuric Acid from Automotive Batteries in the Initial Development of Maize (*Zea Mays L.*)

ABSTRACT

Objective – This study aims to evaluate the technical feasibility of producing ammonium sulfate from industrial residues and to assess its agronomic performance as a nitrogen and sulphur fertiliser for maize cultivation.

Methodology – The research adopted an experimental approach based on controlled pot trials. Ammonium sulfate was produced using residual sulphuric acid from automotive batteries and ammonia liquor from the steel industry. A second formulation was prepared using residual acid and commercial ammonia, while analytical-grade ammonium sulfate (P.A.) was used as a reference. Fertiliser solutions were applied to soil at nitrogen rates ranging from 20 to 120 kg N·ha⁻¹, followed by sowing of maize (*Zea mays L.*). Plant growth and biomass accumulation were subsequently evaluated.

Originality/Relevance – The study addresses a relevant gap concerning the reuse of industrial residues for fertiliser production, contributing to discussions on reducing Brazil's dependence on imported fertilisers. It advances knowledge on alternative nutrient sources aligned with circular economy principles.

Results – Maize plants showed positive responses to all ammonium sulfate sources tested. However, higher application rates resulted in phytotoxic effects, particularly in aerial biomass, while root biomass was negatively affected even at lower nitrogen doses.

Theoretical/Methodological Contributions – The findings contribute to the understanding of nutrient dynamics and toxicity thresholds associated with alternative ammonium sulfate sources, offering methodological insights for evaluating residue-derived fertilisers under controlled conditions.

Social and Environmental Contributions – The proposed approach promotes the valorisation of industrial residues, potentially reducing environmental liabilities and contributing to more sustainable fertiliser supply chains. Additionally, it supports strategies aimed at enhancing agricultural sustainability and fostering a more resilient and autonomous agricultural input supply.

Keywords: Industrial residues. Soil fertility. Crop development. Alternative fertilisers.

Utilización de sulfato de amonio en el suelo producido con licor de amoníaco residual de la industria siderúrgica y ácido sulfúrico residual de baterías automotrices en el desarrollo inicial del cultivo de maíz (*Zea mays* L.)

RESUMEN

Objetivo – Este trabajo tiene como objetivo evaluar la viabilidad técnica de la producción de sulfato de amonio a partir de residuos industriales y analizar su desempeño agronómico como fertilizante de nitrógeno y azufre en el cultivo de maíz.

Metodología – La investigación adoptó un enfoque experimental basado en ensayos controlados en macetas. El sulfato de amonio se produjo utilizando ácido sulfúrico residual proveniente de baterías automotrices y licor amoniacal de la industria siderúrgica. Una segunda formulación se preparó empleando ácido residual y amoníaco comercial, mientras que el sulfato de amonio (P.A.) se utilizó como referencia. Las soluciones fertilizantes se aplicaron al suelo en dosis de nitrógeno que variaron de 20 a 120 kg N·ha⁻¹, seguidas de la siembra de maíz (*Zea mays* L.). Posteriormente, se evaluaron el crecimiento de las plantas y la acumulación de biomasa.

Originalidad/Relevancia – El estudio aborda una brecha relevante relacionada con el reaprovechamiento de residuos industriales para la producción de fertilizantes, contribuyendo a las discusiones sobre la reducción de la dependencia de Brasil respecto a fertilizantes importados. Asimismo, profundiza el conocimiento sobre fuentes alternativas de nutrientes en consonancia con los principios de la economía circular.

Resultados – Las plantas de maíz presentaron respuestas positivas a todas las fuentes de sulfato de amonio evaluadas. Sin embargo, tasas de aplicación más elevadas resultaron en efectos fitotóxicos, especialmente en la biomasa aérea, mientras que la biomasa radicular se vio afectada negativamente incluso en dosis más bajas de nitrógeno.

Contribuciones Teóricas/Metodológicas – Los resultados contribuyen a la comprensión de la dinámica de nutrientes y de los límites de toxicidad asociados a fuentes alternativas de sulfato de amonio, ofreciendo aportes metodológicos para la evaluación de fertilizantes derivados de residuos en condiciones controladas.

Contribuciones Sociales y Ambientales – El enfoque propuesto promueve la valorización de residuos industriales, potencialmente reduciendo pasivos ambientales y contribuyendo a cadenas de suministro de fertilizantes más sostenibles. Además, apoya estrategias orientadas al fortalecimiento de la sostenibilidad agrícola y al fomento de un sistema de suministro de insumos agrícolas más resiliente y autónomo.

Palabras clave: Residuos industriales, fertilidad del suelo, desarrollo de cultivos, fertilizantes alternativos.

1 INTRODUCTION

The maize (*Zea mays L.*) crop is one of the main cereals used for human and animal consumption, as well as a raw material for producing beverages, fuels, and polymers. The United States is the largest producer, followed by China and Brazil, which produced 137 million tonnes in the 2022/23 harvest. According to Prochnow (2007), the maize crop, along with wheat and rice, consumes 60% of all nitrogen used as fertilizer.

Imports of primary macronutrient fertilisers, NPK, have increased from 32% of the total used in 1988 to 80% in 2019, according to data from ANDA (2019), with nitrogen accounting for 35% of this total, primarily sourced from Russia, China, and Belgium (SAE-PR, 2020).

Nitrogen, as a primary macronutrient, is the most consumed nutrient by the maize crop (Nunes et al., 2015). It is important for the vegetative development of the plant, grain productivity, and is essential for the composition of amino acids, proteins, chlorophyll, and enzymes (Malavolta, 2006). According to Sangoi et al. (2014), among the various physiological and anatomical benefits of nitrogen fertilisation in maize are the stimulation of leaf regeneration, delay of leaf senescence, thus maintaining an active photosynthetic area for longer periods, and an increase in leaf area index (LAI), promoting greater CO₂ absorption and element assimilation.

Coke oven gas is a by-product of coal carbonisation in the steel industry. The gas, after generation, is cooled, and one of its by-products is residual ammonium liquor. The production rate is approximately 120 to 130 litres of ammonium liquor per tonne of coal charged (Wright, 2001).

Lead-acid batteries are used in automotive batteries and telecommunications systems due to their high discharge capacity and low production cost. They are constructed using wet technology, employing sulphuric acid (H₂SO₄) in liquid form as the electrolyte. This acid acts as a conductor, transporting electric ions between the positive and negative plates when the battery is being charged or discharged. During battery recycling, the acid is neutralised with an alkaline substance, transforming it into a less harmful residue.

Soils, in general, do not have the capacity to meet the nitrogen demand of maize plants due to their high requirement for the element, making external application necessary to meet the plants' needs. (Delmar and Wietholter, 2004).

The fertilisation of maize with nitrogen fertiliser depends on various factors such as soil physicochemical characteristics, climatic conditions, cultivar used, and crop yield expectations (Rhezali and Lhalali, 2017; Martins, 2013).

According to Coelho (2006), nitrogen is the most limiting mineral element for the development of C4 metabolism crops such as maize, which demand large quantities. Due to the high cost of nitrogen fertilisers and their losses, rational management during the crop cycle is essential for improving productivity and economic sustainability (Filho, 2022). Research is being conducted to reduce nitrogen losses in the soil through the development of new products, but with relative success (Abranches et al., 2018). Additionally, excessive use can promote the pollution of water resources (Santos, 2020) causing serious environmental problems, as observed by Vidal and Neto (2014), who found a high degree of eutrophication in the waters of a reservoir in Northeast Brazil due to its increasing concentration of ammonia.

Sulphur deficiency is observed in soils of maize-producing countries like Brazil (Hitsuda et al., 2008). This has intensified in recent decades due to limited use of sulphur-containing fertilisers, increased extensive agriculture (Scherer, 2001), and depletion of soil organic matter which contains up to 95% of soil sulphur (Sainz Rozas et al., 2011).

Several studies have shown that nitrogen fertilisation combined with sulphur, a secondary macronutrient, has positive interactions improving crop yields (Wilson et al., 2020; Li et al., 2019). According to Vieira (2017), ammonium sulfate is the second most used nitrogen fertiliser in Brazil. It contains about 23% nitrogen but its high production and marketing costs limit widespread use.

Ammonium sulfate fertilizer has several positive characteristics compared to other nitrogen fertilizer sources, such as: not generating toxic ammonia in the soil and consequently avoiding risks of loss through volatilization; a lower leaching effect compared to nitrate, reducing losses and risks of groundwater contamination; a less intense denitrification process; and reduced emissions of NO and N₂O, which are important greenhouse gases (Chien et al., 2011).

In recent decades, especially since the 2000s, global ethanol production has increased significantly due to environmental concerns and the reduction of greenhouse gases, as well as specific government programmes such as the Renewable Fuels Programme in the United States and, in Brazil, the introduction of flex-fuel vehicles. This has stimulated the activities of ethanol plants (ÚNICA, 2003). According to the Renewable Fuels Association (RFA, 2023), the United States is the world's largest ethanol producer, with 59 billion litres per year, almost entirely from maize as raw material. Brazil ranks second, producing 30.5 billion litres (2022/23 harvest), with 85% of this volume derived from sugarcane and 15% from maize (Conab, 2023).

It is projected that by 2030, about 20% of ethanol in Brazil will be maize-based (Neves et al., 2021). The FAO forecasts a global ethanol production increase of 16 billion litres by 2029, half from Brazil (Neves et al., 2021).

According to UNEM (2023), it is estimated that Brazil will reach the mark of 9.65 billion litres of maize ethanol, a growth of 186% compared to the 2020/21 harvest. The 2031/32 corn harvest is expected to reach a production of 149.9 million tonnes of grain, well above the 113.3 million tonnes produced in the 2020/21 harvest.

This study aims to evaluate the ammonium sulfate fertiliser produced using ammonia liquor recovered during the coking process at a steelworks and residual sulphuric acid extracted from spent automotive batteries, compared to ammonium sulfate produced with commercial ammonia and residual acid.

2 METHODOLOGY

The experiment was conducted in the Laboratory of Reuse Studies of Industrial and Mining Residues at the Universidade Federal dos Vales do Jequitinhonha e Mucuri, Teófilo Otoni Campus. Pots with a capacity of 300 mL were used, and the substrate was washed sand with a particle size below 2.8 mm.

Three sources of ammonium sulfate were evaluated by applying them to the substrate to assess the early growth of the maize (*Zea mays* L.) crop. High-purity analytical-grade ammonium sulfate (A.R. grade) and two laboratory-developed ammonium sulfate sources were

used. The first nitrogen fertiliser, ammonium sulfate, was produced using residual sulphuric acid from automotive batteries and commercial ammonia with a concentration of 5.8%. The sulphuric acid was obtained from spent automotive batteries returned for recycling. Due to its high lead contamination, the acid was filtered through a cellulose filter, resulting in sulphuric acid with a lead content below 10 ppm. The acid concentration in the solution was determined by titration in the laboratory using sodium hydroxide, obtaining a value of 8.45%.

After characterizing the concentration of the residual sulfuric acid in the acidic solution, the reaction between the residual sulfuric acid and commercial ammonia was carried out. Two hundred milliliters (200 mL) of 5.8% commercial ammonia were placed in a 500 mL beaker, and the residual sulfuric acid was slowly added, totalling 180 mL. With the constant addition of the sulfuric acid to the commercial ammonia solution, the reaction produced a yellowish/orange-coloured solution that persisted until the entire ammonia had reacted with the commercial sulfuric acid, at which point the medium changed from orange-yellow to transparent. At this point, the titration was stopped, and the ammonium sulfate solution was placed in an oven to dry at 75°C for 72 hours.

The other preparation of ammonium sulfate was carried out by reacting residual sulphuric acid from automotive batteries with ammonia liquor generated in the coking plant of the Usiminas steelworks, located in the municipality of Ipatinga, Minas Gerais. The ammonia liquor collected at the company had an ammonia concentration of 11%. Similar to the previous reaction, 200 mL of ammonia liquor were added to a 500 mL beaker, and the residual sulphuric acid from automotive batteries, totalling 380 mL, was slowly added. The reaction was considered complete when the colour changed from yellow/orange to transparent. After completing the chemical reaction, the produced ammonium sulfate solution was dried in an oven at 75°C for 72 hours.

After drying, white/transparent crystals were formed. The crystals were manually fragmented to facilitate their handling and application. After drying and fragmentation of the ammonium sulfates produced in the laboratory, the materials were weighed on a precision balance and dissolved in distilled water at a concentration of 35.0 grams per litre.

Samples of the laboratory-produced ammonium sulfates were taken for chemical analysis of sulfur, nitrogen, and contaminants. The results are shown in Table 1.

Table 1 - Chemical analysis of nitrogen fertilizers (ammonium sulfates) used in the experiment.

Sample	S	Ammonia - N	Total N	C	Cr	Cu	Cd	Ni	Pb	As	Zn
Unit	%	%	%	%	ppm						
Ammonium Sulfate Usiminas	30.1	0.23	17.4	0.20	10.5	5.93	<1.0	18.9	20.7	<1.0	26.0
Ammonium Sulfate Commercial Ammonia	25.8	0.24	16.8	0.45	10.4	5.16	<1.0	17.0	21.3	<1.0	24.0
Ammonium Sulfate P.A	24.2	-	27.3	-	-	-	-	-	-	-	-

Source: Authors (2025).

In the fertiliser trial, single-cross hybrid maize (*Zea mays* L.) was used. The washed sand used as substrate, after sieving, was added to the pots. Nutrient solutions were then applied to the substrates: 2.0 mL per pot of a solution containing 7.62 g/L potassium chloride (KCl) and 12.8 g/L dibasic calcium phosphate (CaHPO_4), and 2.0 mL per pot of a solution of 55.0 g/L calcium carbonate (CaCO_3) and 22.0 g/L magnesium carbonate (MgCO_3).

The treatments used in the experiment were: control (0 kg/ha), 20, 40, 60, 80, 100, and 120 kg/ha of ammonium sulfate nitrogen fertilisers. The fertilisers were applied to the substrates as solutions prepared with distilled water. The volumes of solution applied to the substrates were determined according to the concentration of each preparation. In this case, each 1,0 mL applied to the substrate of the ammonium sulfate solution corresponded to 10 kg N/ha. The calculation of proportionality was based on the soil volume in the field at 0.2 metre depth per hectare, corresponding to a volume of 2,000 m³, and the pot volume of 300 cm³.

After the solutions were applied to the pots, the maize was sown. Seven seeds were sown per pot, followed by irrigation with 50 mL of water. After germination, two plants per pot were maintained. Irrigation during development was performed as needed. The pots were kept under artificial lighting for 25 days. After this period, the photosynthetic rate of the maize leaves was measured. The two youngest leaves, with the blade fully exposed to light, were evaluated. The reading was performed using the Falker ClorofiLOG chlorophyll meter. After measuring the photosynthetic rate, the plants were cut at the collar region and immediately weighed on a precision balance to determine their fresh weight. The plants were then placed in paper bags and dried in an oven at 75°C for 72 hours to determine their dry weight.

The roots were removed from the substrate by gently washing them under running water. After removal, the roots were kept in the shade for six hours to lose excess moisture from the washing water. After this period, the roots were weighed on a precision balance to determine their fresh weight. They were then placed in paper bags and dried in an oven at 75°C for 72 hours to determine their dry weight.

The variables analysed in the plants were: fresh and dry weight of the aerial part, roots, and total fresh weight, the shoot-to-root fresh weight ratio and photosynthetic rate. A 3x7 factorial design was used, and the experiment was completely randomised with four replications. ANOVA was used to test the hypotheses for the analysed variables with $P < 0.05$, and the Tukey test was applied to verify differences between means. Regression analysis was also performed on the obtained data.

3 RESULTS AND DISCUSSION

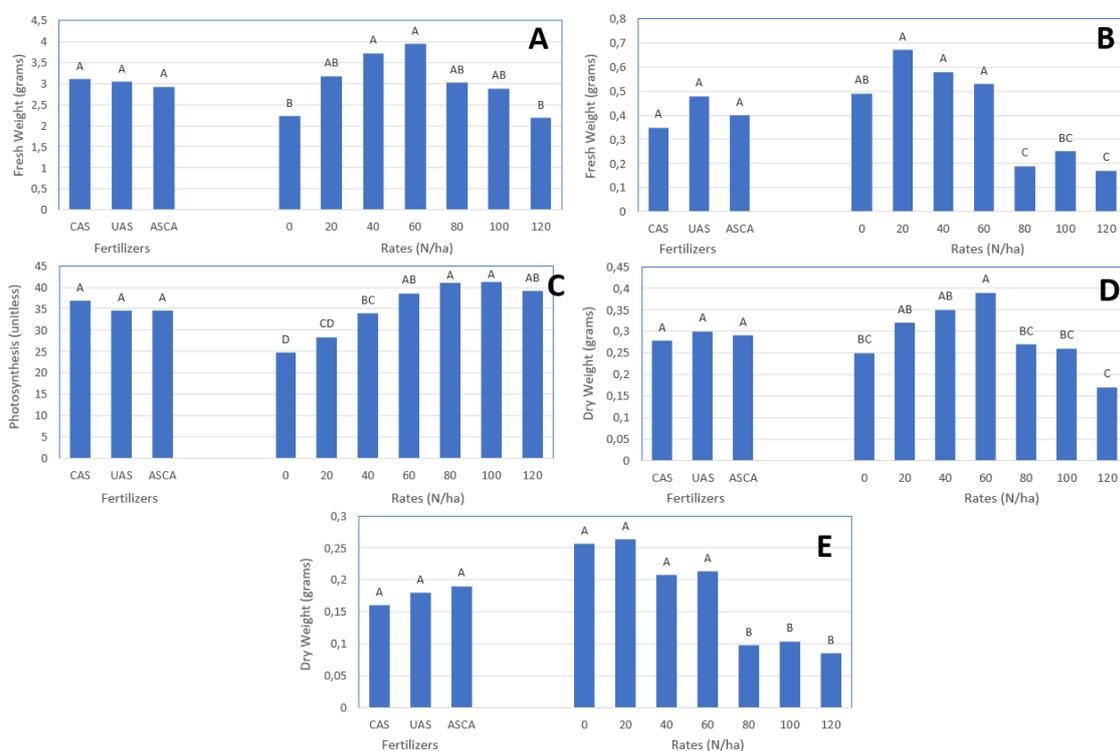
No statistically significant differences were observed among the evaluated ammonium sulfates in all analysed variables of the maize plants (fresh and dry weight of the aerial part and roots, and photosynthetic rate) (Figures 1A, 1B, 1C, 1D and 1E), regardless of the amount applied to the soil. These results indicate that the ammonium sulfate produced with residual coking liquor combined with residual sulphuric acid has a positive effect on plant development, similar to purified commercial ammonium sulfate. The same behaviour was observed with ammonium sulfate produced with commercial ammonia and sulphuric acid.

Regarding the doses of ammonium sulfate applied to the soil, regardless of the sources used, the behaviour in relation to the fresh weight of the aerial part of the plants (Figure 1A) showed better performance at doses of 40 and 60 kg N/ha, superior to the control treatment (0 kg N/ha) and the highest dose (120 kg N/ha). In the first case, this was probably due to the absence of nitrogen in the substrate, and in the second case, due to excess causing toxicity in the plants. In the case of fresh root weight (Figure 1B), the lower nitrogen doses showed visual gains in fresh mass but were not statistically superior to the control treatment. Nitrogen fertilizer doses of 80 kg N/ha and above (up to 120 kg N/ha) resulted in a significant reduction in root development.

Regarding the photosynthetic rate (Figure 1C), the physiology of the photosynthetic process did not show toxic effects from excess nitrogen as observed in the fresh weight development of the aerial part and roots, with photosynthetic activity stabilising, probably due to reaching its maximum peak.

The dry weight performance of the aerial part (Figure 1D) and roots (Figure 1E) of the maize plants was similar to that seen for the fresh weight of the plants.

Figure 1 – Fresh weight of shoot (A); fresh weight of the roots (B); photosynthetic rate (C); dry weight of the shoot (D) and dry weight of the roots (E) of maize plants grown in substrate with different sources and doses of ammonium sulfate fertiliser.



Source: Authors (2025).

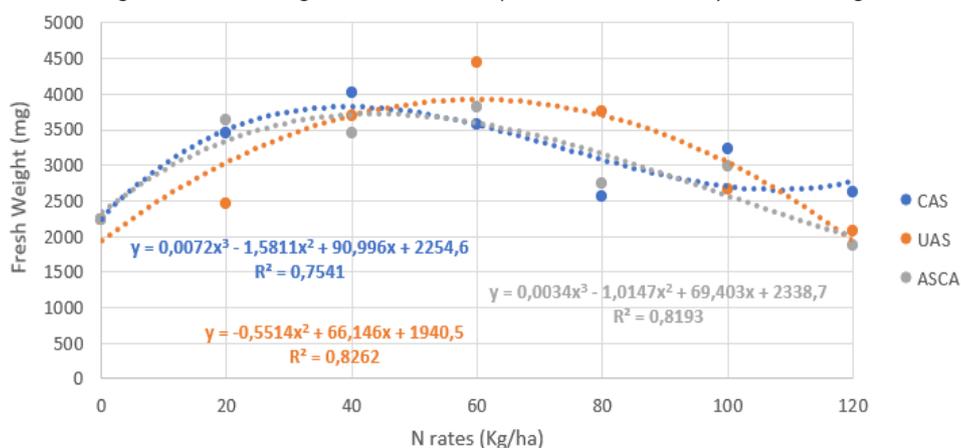
The three nitrogen sources initially tested in the development of the maize crop showed significant effects according to the quantities used. In Figure 2, the behaviour of the fresh weight development of the maize aerial part is shown according to the doses used and the nitrogen sources. Commercial ammonium sulfate (CAS) and ammonium sulfate produced by the reaction of residual sulphuric acid from automotive batteries and commercial ammonia (ASCA)

stimulated the fresh weight performance of the maize shoot more significantly at the lower doses (20 kg N/ha), with increases of about 50% compared to the control (0 kg N/ha) in the adjusted regression model. In these treatments, the peak fresh mass production of the shoot was approximately 3.75 g/pot with the use of 40 kg N/ha, about 60% higher than the treatment without nitrogen source. From this dosage of CAS and ASCA sources, the aerial part performance showed a significant decline up to the highest doses evaluated (120 kg N/ha).

When comparing the performance results of maize plants under the effect of the ammonium sulfate fertilizer produced by the reaction of residual sulfuric acid from automotive batteries with residual liquid ammonia from a steelmaking coke plant, some differences are observed. At the lowest tested dose of Usiminas Ammonium Sulfate (UAS) (20 kg N/ha), the maize plants showed lower performance in terms of shoot fresh mass compared to the other nitrogen fertilizer sources, even though the regression model adjustment indicated a similar growth rate, but a performance approximately 17% lower than the other treatments. Despite these lower values of fresh mass production, at the dosage of 40 kg N/ha, the performance of plants treated with UAS was similar to that of plants treated with the other nitrogen sources (CAS and ASCA).

While in treatments using CAS and ASCA the peak production of shoot fresh mass of maize plants occurred at a dose close to 40 kg N/ha, in the case of UAS the adjusted peak of maximum maize plant productivity was achieved at a dose of 60 kg N/ha, with no indication of any signs of toxicity from the components present in the nitrogen fertilizer prepared with residual ammonia liquor up to this tested dose. In this case, considering the values adjusted by the regression model, the shoot fresh mass production of maize was 100% higher than the control treatment (without nitrogen fertilizer) and showed a superior plant response compared to the other treatments. Beyond this dosage, a gradual decline in plant performance occurs with increasing doses of the applied nitrogen fertilizer (UAS) up to the dosage of 120 kg N/ha.

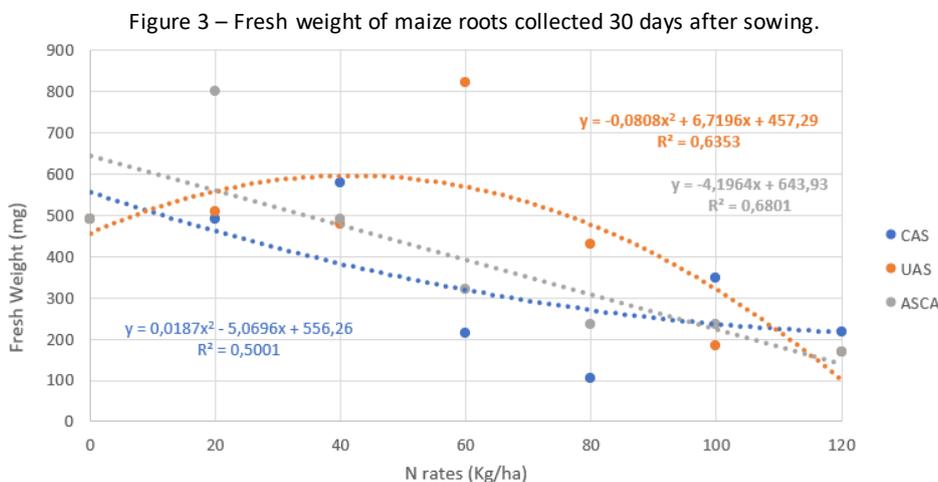
Figure 2 – Fresh weight of maize shoot plants collected 30 days after sowing.



Source: Authors (2025).

The behavior of the maize plant roots showed specific responses to each nitrogen fertilizer tested. Figure 3 shows that, although they followed the same trend in the regression model curve adjustment, the roots exhibited distinct behavior at the same application doses of the nitrogen fertilizers, with continuous declines in fresh mass as the nitrogen fertilizer doses

increased, reaching minimum performance at the dosage of 120 kg N/ha—a reduction in root fresh mass of approximately 60% in the CAS and ASCA treatments.



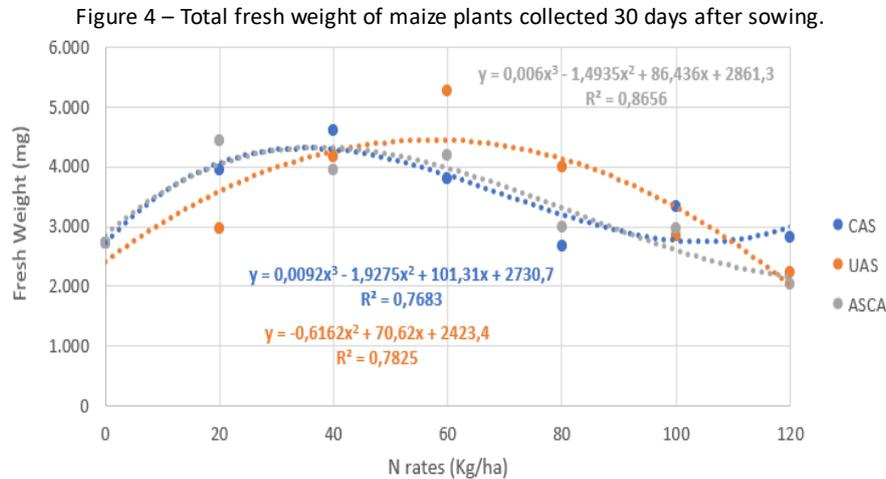
Source: Authors (2025).

The UAS nitrogen fertilizer promoted good maize root performance, differing from the other tested fertilizers (SAC and SAAC). As shown in the regression curve adjustment, the peak production of maize root fresh mass occurred around the dose of 40 kg N/ha, with values approximately 30% higher than the control treatment (0 kg N/ha). Beyond this dosage, a decline in root fresh mass production was observed until reaching a minimum at the highest UAS fertilizer dosage (120 kg N/ha). In this case, the reduction in root performance was approximately 80% compared to the 0 kg N/ha dose. For all nitrogen fertilizers tested, a toxic effect of these compounds on the soil was observed when applied in high quantities. The use of sandy substrate in the trial, with minerals of low CEC and AEC, allowed greater interaction of mineral elements with the soil solution and directly with the plant roots. Albornoz (2016) states that excessive nutrient supply, such as nitrogen, can lead to negative effects on plant growth due to nutritional imbalance.

The total fresh weight of maize plants (shoot + root) (Figure 4) showed results with a wet mass accumulation trend similar to the behavior of the individual plant parts across all treatments. The CAS and ASCA treatments induced maximum fresh mass accumulation in plants between 30 and 40 kg N/ha, with significant declines as the dosage of applied nitrogen fertilizer increased. Compared to the treatment without nitrogen fertilizer application (0 kg N/ha), the production increase exceeded 50%. At the maximum tested dosages of CAS and ASCA fertilizers, the fresh mass production of maize plants was similar to the control treatment.

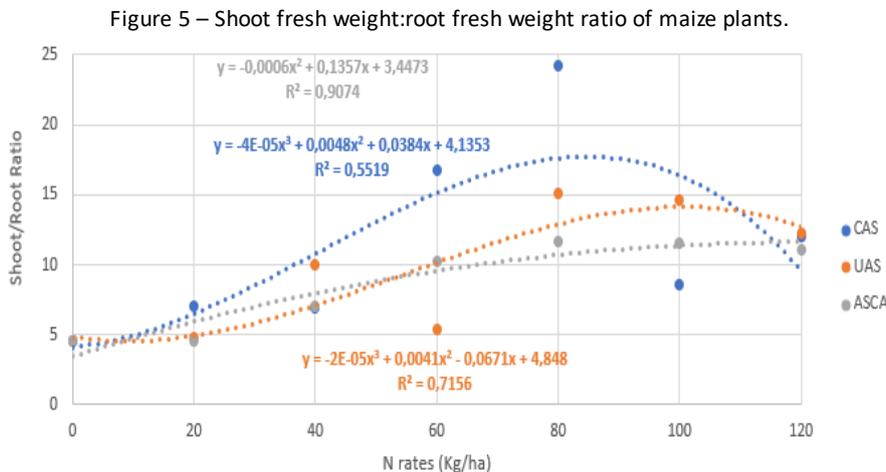
The performance of maize plants in the UAS (Usiminas Ammonium Sulfate) treatment differed somewhat from the other treatments. The plant response in total fresh mass accumulation was observed up to the dose of 40 kg N/ha, where values became equivalent. Beyond this dosage (40 kg N/ha), the maize plant mass accumulation continued to increase until the dosage of 60 kg N/ha. Compared to the control treatment (0 kg N/ha), the increase was approximately 75%, based on the regression curve adjustment. From the dosage of 60 kg N/ha onward, the fresh mass accumulation of maize plants declined, likely due to toxicity from excess

soluble mineral elements, until reaching a total fresh weight similar to the control treatment at the applied dosage of 120 kg N/ha.



Source: Authors (2025).

Figure 5 shows distinct behavior in the shoot-to-root fresh weight ratio among the treatments.



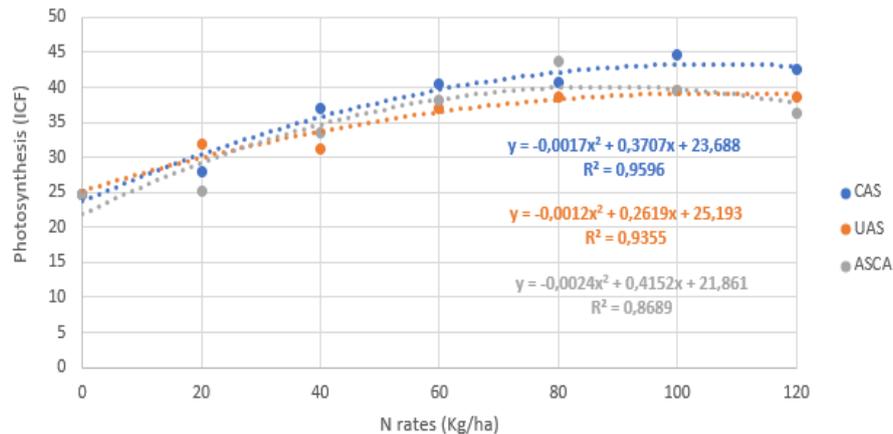
Source: Authors (2025).

In the CAS (Commercial Ammonium Sulfate) treatment, the ratio increased significantly up to the dosage range of 80–90 kg N/ha, with a peak shoot-to-root ratio of 18:1 according to the regression model adjustment. Considering that reductions in plant fresh mass production began at a dosage of 40 kg N/ha, the detrimental effects of excessive nitrogen—beyond the plant’s optimal response level—appeared to impact the root system more severely. This is evidenced by the decline in fresh mass production for both shoots and roots from the dose of 40 kg N/ha up to the applied dose of 80–90 kg N/ha. Beyond this point up to the maximum dose of 120 kg N/ha, damage to the shoot also intensified, leading to sharp declines in the ratio values. In the UAS and ASCA treatments, a slow and steady increase in the ratio was observed, even at higher doses, reaching values between 12:1 and 13:1. This indicates a negative

interference of the fertilizers in the soil at higher doses, affecting the roots more intensely compared to the shoot.

In Figure 6, the photosynthetic rate of maize plant leaves was evaluated during the plant collection period.

Figure 6 – Evaluation of photosynthesis in maize plants.



Source: Authors (2025).

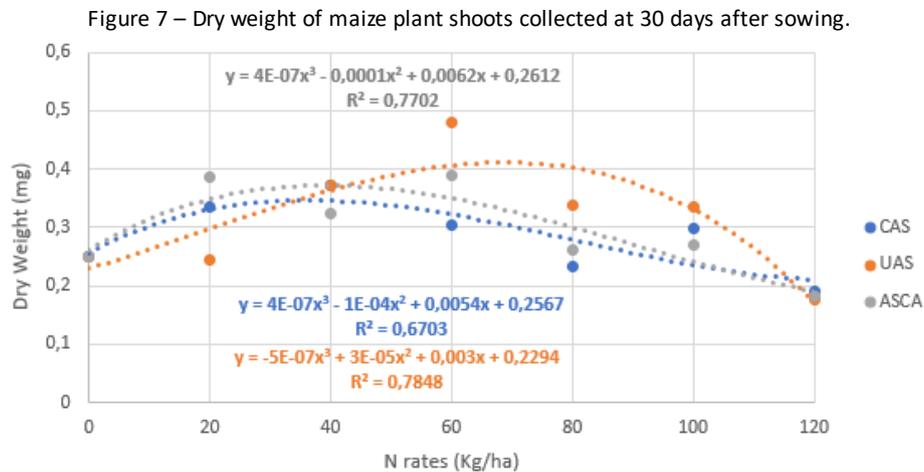
A similar behavior was observed for the three treatments assessed, where the rate increased with continuous rises in fertilizer dosage up to 80 kg N/ha. Beyond this dosage, values stabilized up to the dose of 120 kg N/ha. These increasing photosynthetic rates, even amid declining maize fresh mass production, indicate that after reaching peak photosynthetic activity, the plants maintained the rate. However, the excess nitrogen in the plant — lacking physiological or photosynthetic function — induced toxicity, stimulating photorespiration and reducing fresh mass production, despite the photosynthetic rate being at its peak. Rhezali and Lahlali (2017) described a strong correlation between nitrogen content and the photosynthetic activity of leaf cells, emphasizing the importance of this element in improving maize crop yield. Pagani and Echeverria (2011) noted that increased photosynthesis results from higher chlorophyll concentrations in maize leaves.

Bullock and Anderson (1998) stated that chlorophyll content readings are a precise indicator of nitrogen levels during maize developmental stages, and according to Zebarth et al. (2002), there is a significant correlation between leaf N content and chlorophyll content, considering that 50%–70% of the plant's total nitrogen is found in enzymes associated with the chloroplasts.

Dry weight of plants may exhibit behaviour slightly different from fresh weight. In shoots, this is due to water content at the time of cutting, and in roots, due to the process of washing and rapid air-drying to remove excess moisture.

The dry weight of maize plant shoots (Figure 7) showed performance behaviour relative to treatments similar to fresh weight. In treatments using the nitrogen fertilizers CAS (Commercial Ammonium Sulfate) and ASCA (Ammonium Sulfate produced with commercial ammonia), maximum dry mass production was observed between doses of 20 and 40 kg N/ha. The highest values were seen in the ASCA treatment, where peak production was approximately 45% higher than the control treatment (0 kg N/ha), based on the regression curve adjustment.

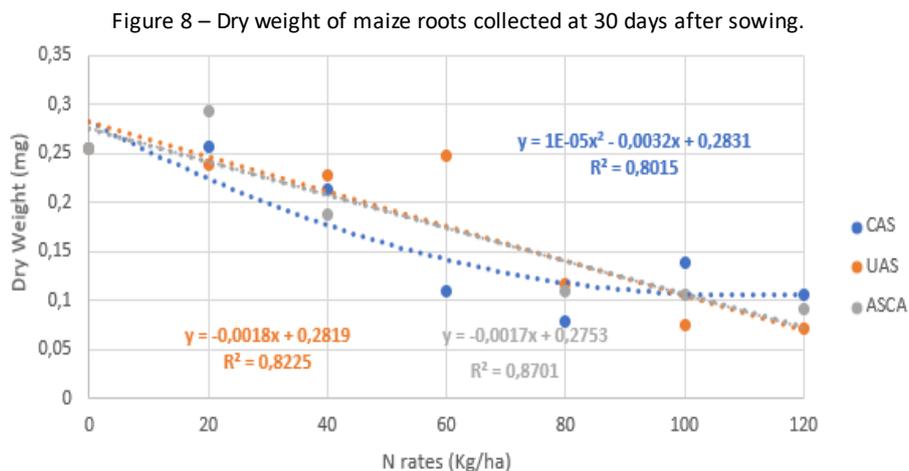
Beyond the dosage of 40 kg N/ha, plant dry mass decreased until values fell below those of the control treatment at the application of 120 kg N/ha for both treatments (CAS and ASCA).



Source: Authors (2025).

Regarding the treatment using nitrogen fertilizer ammonium sulfate produced with residual ammonia liquor from steelmaking (UAS), plant performance was inferior to other treatments up to the dosage of 40 kg N/ha. Despite this, dry matter development continued to increase with higher fertilizer doses, reaching peak production between 60 and 80 kg N/ha with values approximately 80% higher than the control treatment (0 kg N/ha) and other treatments. With further increases in soil fertilizer dosage, shoot dry mass production decreased to values below the control treatment at 120 kg N/ha and similar to other treatments. Souza and Fernandes (2006) cite studies demonstrating that nitrogen in the ammonium form can become toxic to plants by interfering with their morphophysiology. This toxicity may induce symptoms such as dry and curled leaves, resulting from increased resistance to water flow. Chlorosis and leaf necrosis can also indicate ammonium toxicity.

The roots exhibited distinct behaviors depending on the treatment and dosage applied (Figure 8). Treatments using CAS and UAS resulted in continuous declines in maize root development up to the maximum applied dosage of 120 kg N/ha. This was likely due to the closed environment (pots), which prevented fertilizer loss and induced the plants to develop smaller root masses as nutrients became more readily available in the substrate. At higher fertilizer doses, toxicity effects may have occurred, also reflecting on shoot development. In the treatment using different doses of ASCA fertilizer, the roots maintained some stability in dry mass production during the initial phase but also showed significant declines as fertilizer application rates increased, ultimately resulting in dry mass values and production levels similar to the other treatments.



Source: Authors (2025).

Restrictions on maize plant development at higher doses of ammonium sulfate were observed in the trials. Similar results regarding protein production in maize were reported by Farinelli and Lemos (2012), where the highest dose of nitrogen applied to the soil (150 kg/ha) did not increase the crude protein content of the plants. Organomineral nitrogen fertilizers are salts that, when water is added to the soil, increase the electrolyte concentration of the soil solution, raising osmotic pressure and inhibiting water absorption by plants (Taiz and Zeiger, 2006). Ammonium sulfate also reduces the pH of the soil at the application site, compromising plant development (Vitti et al., 2002), a fact that may explain the reduced performance of maize roots at all tested doses and of the shoot beyond certain dosage levels.

Coelho (2007) states in his work that the average amount of nitrogen used in maize cultivation in Brazil is 60 kg N/ha. To achieve high yields, this value can reach 100 kg N/ha for top-dressing alone in non-irrigated cultivation, and between 120 and 160 kg N/ha in irrigated cultivation (Amaral Filho, 2005; Pavinato et al., 2008). All cited results were obtained in field conditions where losses of the element due to environmental and management factors are considered. In this experiment, maize was grown in a closed system with sandy substrate without fertilizer losses, a factor that likely induced plant toxicity responses even at lower doses.

4 CONCLUSIONS

Based on the results obtained in this experiment, it can be concluded that the three sources of ammonium sulfate improved the performance of maize shoots up to a certain dose, with toxic effects occurring beyond the plant maximum production dose. With Usiminas ammonium sulfate, the peak shoot biomass production occurred at a higher dosage than the other sources, indicating lower toxicity, although a significant decline in maize shoot production was observed after the maximum productivity dose.

In relation to the roots, a distinction was observed between dry weight and wet weight, indicating variations in the dynamics of water accumulation and retention in the roots. Overall, however, the results demonstrated that toxicity was more intense in the roots than in the shoots, regardless of the ammonium sulfate source.

Even at the highest application rates, where toxicity symptoms and biomass reduction were evident, photosynthetic activity remained elevated, with peak values observed near the upper ammonium sulfate doses. These findings indicate that, under the experimental conditions, photosynthetic performance persisted at levels where biomass production was already impaired.

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DECLARAÇÕES

CONTRIBUIÇÃO DE CADA AUTOR

Ao descrever a participação de cada autor no manuscrito, utilize os seguintes critérios:

- **Concepção e Design do Estudo:** Alexandre Sylvio Vieira da Costa e Marcos Cordeiro Fonseca conceberam a ideia central do estudo, definiram os objetivos e delineararam a abordagem metodológica adotada.
- **Curadoria de Dados:** Alexandre Sylvio Vieira da Costa foi responsável pela organização, verificação e validação dos dados experimentais, assegurando sua qualidade e consistência.
- **Análise Formal:** Alexandre Sylvio Vieira da Costa realizou as análises formais dos dados, aplicando os métodos analíticos e estatísticos necessários à interpretação dos resultados.
- **Aquisição de recursos:** Marcos Cordeiro Fonseca contribuiu para a obtenção dos recursos para testes e institucionais necessários à execução do estudo.
- **Investigação:** Alexandre Sylvio Vieira da Costa conduziu a investigação experimental, incluindo a execução dos ensaios e a coleta dos dados.
- **Metodologia:** Alexandre Sylvio Vieira da Costa desenvolveu, aplicou e ajustou as metodologias empregadas no estudo, com contribuições conceituais de Marcos Cordeiro Fonseca.
- **Redação - Rascunho Inicial:** Alexandre Sylvio Vieira da Costa elaborou a primeira versão do manuscrito.
- **Redação - Revisão Crítica:** Marcos Cordeiro Fonseca revisou criticamente o manuscrito, contribuindo para o aprimoramento da clareza, coerência e fundamentação técnica do texto.
- **Revisão e Edição Final:** Alexandre Sylvio Vieira da Costa e Marcos Cordeiro Fonseca realizaram conjuntamente a revisão final e os ajustes necessários para atender às normas da revista.
- **Supervisão:** Alexandre Sylvio Vieira da Costa supervisionou o desenvolvimento do trabalho, garantindo a qualidade científica e a coerência global do estudo.

DECLARAÇÃO DE CONFLITOS DE INTERESSE

Nós, **Alexandre Sylvio Vieira da Costa e Marcos Cordeiro Fonseca**, declaro(amos) que o manuscrito intitulado "**Use of Ammonium Sulfate in Soil Produced with Residual Ammonia Liquor from the Steel Industry and Residual Sulfuric Acid from Automotive Batteries in the Initial Development of Maize (Zea Mays L.)**":

1. **Vínculos Financeiros:** Não possui vínculos financeiros que possam influenciar os resultados ou interpretação do trabalho ou "Nenhuma instituição ou entidade financiadora esteve envolvida no desenvolvimento deste estudo".
2. **Relações Profissionais:** Não possui relações profissionais que possam impactar na análise, interpretação ou apresentação dos resultados ou "Nenhuma relação profissional relevante ao conteúdo deste manuscrito foi estabelecida".
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