AGRONOMY (AGRONOMIA)

Volatilized N-NH$_3$, N, and C stocks in soil after green manuring of coffee crops

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ABSTRACT: Volatilization of nitrogen ammonia (N-NH$_3$) and rapid incorporation of mineralized nitrogen (N) into soil organic matter fractions can reduce the efficiency of green manuring. The objective of this study was to characterize N-NH3 loss through volatilization and the changes in carbon (C) and N stocks in soil fertilized with Crotalaria juncea and ammonium sulfate during coffee plant cultivation. The N-NH$_3$ collectors were installed and evaluated at 1, 2, 3, 4, 5, 6, 9, 12, 15, 18, 25, 32, 67 and 123 days after their installation. Total organic carbon (TOC) and total nitrogen (TN) in the 0–5- and 5–10-cm soil layers were evaluated at 0, 60, 120, 180, 240, and 300 days after fertilization. After 9 days, 20.83 kg ha^{-1} (N-NH$_3$) had volatilized from the 41.88 kg ha^{-1} observed at the end of the evaluation period. The TOC stock in both soil layers was 5.81 Mg ha^{-1}, and after 300 days, the stocks were 8.84 and 8.36 Mg ha^{-1} for the 0–5 and 5–10 cm layers, respectively. The TN stock in the 0–5 cm layer increased from 1.74 to 1.98 Mg ha^{-1} over 60 days, and then decreased. There was no time dependency in the TN stock in the 5–10 cm layer. Volatilization of N-NH$_3$ was rapid but low. The increase in TOC stocks was not followed by an increase in TN stocks.

Key words: ammonium; green manure; organic matter

N-NH$_3$ volatilizado e estoques de N e C de solo após adubação verde de cafeeiro

RESUMO: A volatilização de N-NH$_3$ e a rápida incorporação do N mineralizado em frações da matéria orgânica do solo podem reduzir a eficiência da adubação verde. O objetivo do trabalho foi caracterizar as perdas por volatilização de N-NH$_3$ e as modificações nos estoques de C e N em solo cultivado com cafeeiro e adubado com Crotalaria juncea e sulfato de amônio. Foram instalados coletores de N-NH$_3$, procedendo-se com avaliações aos 1, 2, 3, 4, 5, 6, 9, 12, 15, 18, 25, 32, 67 e 123 dias após a sua instalação. Os estoques de carbono orgânico total (COT) e nitrogênio total (NT) do solo foram avaliados, nas camadas de 0-5 e 5-10 cm, aos 0, 60, 120, 180, 240 e 300 após as adubações, sendo a primeira amostragem antes das adubações. Aos nove dias já haviam sido volatilizados (N-NH3) 20,83 kg ha^{-1} dos 41,88 kg ha^{-1} observados no final do período de avaliação. O estoque de COT em ambas as camadas era de 5,81 Mg ha^{-1} e após 300 dias os estoques foram de 8,84 e 8,36 Mg ha^{-1}, respectivamente para as camadas 0-5 e 5-10 cm. Já o estoque NT na camada 0-5 cm aumentou até os 60 dias, passando de 1,74 para 1,98 Mg ha^{-1}, e em seguida apresentou redução. Não houve efeito do tempo no estoque de NT na camada 5-10 cm. A volatilização de N-NH$_3$ é rápida, porém é baixa. O aumento nos estoques de COT não foi acompanhado por aumentos nos estoques de NT.

Palavras-chave: amônia; adubo verde; matéria orgânica

Introduction

The use of synthetic fertilizers increases agriculture production costs and may have negative impacts on the environment. Nitrogen fertilizers are commonly used in agriculture and require fossil fuels for their acquisition, making their use unsustainable (EFMA, 2000). Therefore, the study of biological processes that target crop nutrition is fundamental and green manure is an interesting alternative to nitrogen fertilizers. The potential benefits to the agroecosystem of green manure are widely known; with this practice, it is possible to increase soil organic matter (SOM) (Araújo et al., 2014) and promote beneficial physical, chemical, and biological properties of soil (Wolf & Wagner, 2005; Yang et al. 2012; Araújo et al., 2014).

Generally, N is the most required nutrient in production systems and legumes can supply this part of this demand (Araújo et al., 2014). However, despite the potential for green manuring to increase SOM and N in the system, its positive effects on crop growth and/or production are not always found when the objective is improving nitrogen nutrition in crops, especially for coffee (Partelli et al., 2011; Moreira et al., 2014).

Several factors may influence crop responses to green manure, such as the species used as the fertilizer, intercropping time, crop area history, soil properties, and allelopathic effects (Alfaia, 1997; Partelli et al., 2011; Carvalho et al., 2014; Moreira et al., 2014). Another factor that may influence the efficiency of green manuring is the volatilization of N-NH$_3$ (Janzen & McGinn, 1991; Ruijter et al., 2010). In perennial crops such as coffee, application of green manure is superficial because its incorporation into the soil may cause damage to the roots and increase the possibility of N losses through volatilization (Ruijter et al., 2010). Studies have shown that N loss by N-NH$_3$ volatilization from organic waste may be important, especially if the material is not incorporated (Ruijter et al., 2010; Shan et al., 2015).

If N-NH$_3$ volatilization losses are rapid, it indicates rapid decomposition and mineralization of the organic residue, which may be reflected in the measurement of soil organic matter (SOM). Thus, because the relationship between SOM, C, and N is known (Lovato et al., 2004; Gauder et al., 2016), the importance of quantifying soil C and N when applying green manure and mineral fertilizers is important to validate. The objective of this study was to characterize N-NH$_3$ volatilization loss and the N and C stock dynamics in soil fertilized with *Crotalaria juncea* and ammonium sulfate during coffee cultivation.

Material and Methods

The experiment was carried out in a protected environment in an area at the Department of Plant Science of the Federal University of Viçosa (UFV), Viçosa, Minas Gerais state (20°45'14"S, 42°52'53"W and 650 m altitude) from January 7th to November 20th, 2013. The experimental unit consisted of a 150 dm^3 and 0.59 m^2 upper area that contained a Catuaí 44 cultivar coffee plant (*Coffea arabica* L.) that was two months old at transplanting. The experiment was performed in five replicates that were evaluated over time.

The soil used in the study is classified as Oxisol that is clay-sand in texture and originates from a previously cropped area. The soil was collected from the field and was filled in the cultivation box such that soil measurements could be taken from the 0–20-cm and 20–40-cm layers. The soil was not sifted. Amendments were made according to Guimarães et al. (1999) and to the chemical and physical characteristics of the collected soil. Chemical and physical characterization of the soil was performed according to Embrapa (2011). The results of the chemical analysis were for the 0–20-cm and 20–40-cm layers are listed in Table 1.

The cultivation boxes were irrigated to maintain soil moisture between 70-80% of their field capacity, which was measured by the ECH$_2$O dielectric sensor. The bottom of the boxes was filled with expanded clay to allow the drainage of the excess water and subsequent return of percolated water to the boxes.

Table 1. Chemical and physical characteristics of soil matter.

Chemical characteristics	Depth (cm)	
	0-20	20-40
pH H$_2$O	5.94	5.71
P (mg dm^{-3})	36.00	5.10
K (mg dm^{-3})	165.00	66.00
Ca^{2+} (cmol$_c$ dm^{-3})	3.20	2.57
Mg^{2+} (cmol$_c$ dm^{-3})	0.75	0.48
Al^{3+} (cmol$_c$ dm^{-3})	0.00	0.00
H+Al (cmol$_c$ dm^{-3})	2.90	3.90
SB (cmol$_c$ dm^{-3})	7.51	4.37
t (cmol$_c$ dm^{-3})	7.51	4.37
T (cmol$_c$ dm^{-3})	11.41	7.27
V (%)	65.80	60.10
N (dag kg^{-1})	0.14	0.12
OM (dag kg^{-1})	3.23	2.45
P-rem (mg L^{-1})	34.00	31.60
Zn (mg dm^{-3})	4.97	1.49
Fe (mg dm^{-3})	96.20	65.20
Mn (mg dm^{-3})	89.80	49.30
Cu (mg dm^{-3})	3.22	1.68
S (mg dm^{-3})	5.40	7.80
Physical characteristics	**0-5**	**5-10**
Soil density (g cm^{-3})	1.10	1.01
Thick sand (g kg^{-1})	270.30	274.70
Fine sand (g kg^{-1})	244.90	239.90
Silt (g kg^{-1})	130.00	124.30
Clay (g kg^{-1})	354.80	361.10
Textural class	Clay-sandy	Clay-sandy

pH in H$_2$O, (1:2.5); P, K, Fe, Zn, Mn, Cu: extracted with Mehlich-1; Ca, Mg, Al: extracted with KCl 1 mol L^{-1}; H + Al, extracted with calcium acetate 0.5 mol L^{-1} - pH 7.0; S: extracted with monocalcium phosphate in acetic acid; SB = Exchangeable Bases Sum; t = Effective Cation Exchange Capacity; T = Cation exchange capacity at pH 7.0; V = Base Saturation Index; OM = organic matter by the Walkley & Black method (total organic carbon × 1.724). P-rem = remaining Phosphorus; N: Kjeldahl distillation. Soil Density: volumetric ring method; Sand, silt and clay: mechanical agitation and chemical dispersion with sieving and sedimentation separation (pipette method). All methods were used according to Embrapa (2011).

Prior to transplanting coffee seedlings, 65 g P_2O_5 (50% simple superphosphate and 50% natural reactive phosphate) were applied to the soil in each box. Nitrogen fertilization was applied as 6 g N per plant (60% via ammonium sulphate (SA) and 40% as arial dry matter of *Crotalaria juncea*.) A recovery efficiency of 50% of mineral N was considered, which implied in the application of 7.2 g box^{-1} N (122 kg ha^{-1} N). For sunn hemp residues, the adopted recovery efficiency was 25%, resulting in a dose of 9.6 g box^{-1} N (162.7 kg ha^{-1} N). The amount of applied residues corresponded to 280 g box^{-1} (4.75 t ha^{-1}) of the fractioned sunn hemp aerial material. The legume had a concentration of 3.4% N in its dry matter. After coffee transplantation, plants were fertilized with 10 g K_2O per plant over four applications, using KCl as the source.

In the first-year after plant fertilization, 15 g N per plant were applied using AS and sunn hemp sources. The N fertilizer recovery efficiency led to doses of 18 g N per plant via AS and 24 g N per plant via sunn hemp residues. Chemical nitrogen fertilization was split in three equal applications, while sunn hemp residues were applied at one time to the last AS plot. However, in this study, mineral fertilization (12 g box^{-1} of N) was only applied to the first two plots.

Legume production

The legume was produced in a greenhouse using 150-dm^3 boxes filled with medium sieved sand and vermiculite in a 1:1 ratio. Sowing was done in October at the ratio of 40 seeds per linear meter, with 50 cm between rows. After seedling emergence, plots were thinned in order to obtain 50 sunn hemp plants per box. The legume was grown until flowering, when it was cut at 77 d after sowing. The material was then dried in the shade, fragmented, and stored in raffia bags until needed. The sunn hemp dry matter was used for the calculation of the applied fertilization.

N-NH$_3$ volatilization

Measurement of N-NH$_3$ began soon after the distribution of the legume mass on the surface of the coffee plant boxes. The N-NH$_3$ collectors were installed and classified as semi-open or static. The collectors were made of PVC pipes of 20 cm diameter and 60 cm in height, and were inserted 5 cm in the soil. These tubes contained two foam disks soaked with a 1 M H_2SO_4 solution made in 3% glycerol; one was to capture volatilized N-NH$_3$ and another to prevent environmental N-NH$_3$ contamination. Inside these tubes, the legume mass was added at the same proportion at that applied to the box.

The volatilized N-NH$_3$ was collected by changing the two foam disks in each collector 1, 2, 3, 4, 5, 6, 9, 12, 15, 18, 25, 32, 67, and 123 days after installation. The lower foam disks were packed in plastic bags and stored in a refrigerator until extraction of $(NH_4)_2SO_4$. The other disk was discarded during each exchange.

To extracted $(NH_4)_2SO_4$ from the foam disks, water in vacuum was produced for two minutes using a Büchner funnel coupled to a kitasate and vacuum pump. The extracted solution was collected and stored in glass flasks under refrigeration until further analysis.

Total N content was determined by distillation using the Kejldahl method (Tedesco et al., 1995) in 10 M NaOH, followed by titration with 0.05 M HCl standardized in H_3BO_3 solution plus an indicator.

Total organic soil C and N stocks

In order to determine the variation in total organic C (TOC) and total N (TN) in the soil, soil samples were collected from 0–5-cm and 5–10-cm layers; the first sample was collected before application of legume and AS fertilizers. Another five samples were collected every two months after the application of the legume fertilizer at 60, 120, 180, 240, and 300 days.

Soil samples were collected with a probe-type auger at six areas (simple samples) per box, forming a sample composed by repetition. The holes produced from the sampling were closed and marked with sticks so that future collections were taken from the same point. Soil samples were then dried in the shade and passed through a 2-mm sieve.

Soil TN measurements were obtained by the same method used for the determination of N-NH$_3$ (Tedesco et al., 1995), using wet oxidation adapted from Tedesco et al. (1995).

Soil stocks of TOC and TN (t ha^{-1}) were calculated with the following formula: TOC or TN stocks = TOC or TN content (g kg^{-1}) × Ds × E/10; where Ds is soil density at the depth (kg dm^{-3}), and E is the soil layer thickness (cm).

Statistical analysis

The results were submitted to analysis of variance (ANOVA) and then evaluated by means of regression (p < 0.05). Different models were tested by regression analysis, and models based on the significance of each of the coefficients, on the value of the coefficient of determination, and that could explain the phenomenon in question. Sisvar software was used to aid in the analysis.

Results and Discussion

N-NH$_3$ volatilization

The volatilization of N-NH$_3$ (Figure 1) had a variable behavior until the 18th day, with high values at the beginning of the evaluation, reduced values until the 12th day, and increased values between the 15th and 18th days. We measured the lowest N-NH$_3$ volatilization after 32 days (Figure 1). There were two peaks of volatilization that have already been reported for organic fertilizer usage (Janzen & McGinn, 1991; Shan et al., 2015), and can be explained by the differential decomposition between more labile and more recalcitrant fractions of the material (Diniz et al., 2014). However, notably, this biphasic behavior of N-NH$_3$ volatilization from organic fertilizers can be compared. Several factors affect volatilization values, such as the material used (i.e. N content and C/N ratio) and environmental conditions (i.e. temperature, soil moisture, initial pH, and soil type).
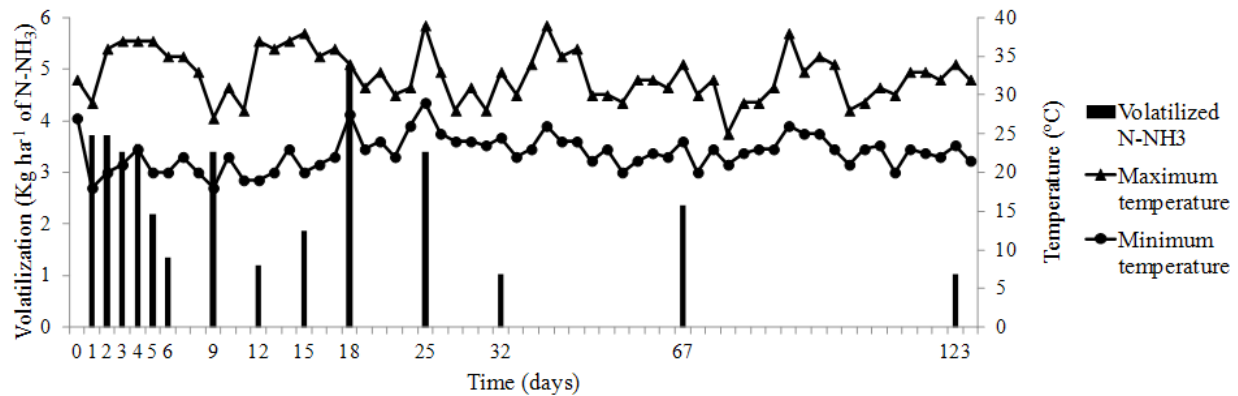
Figure 1. Air temperature in the protected experimental growth environment and volatilized N-NH$_3$ measured in coffee plant cultivation boxes supplied with superficial application of sunn hemp organic matter.

The initial N-NH$_3$ volatilization behavior was similar to those reported for mineral fertilizers, with the highest volatilization peaks occurring just hours after fertilization (Rochette et al., 2009; Soares et al., 2012). However, the volatilization peaks we observed on the 15th and 18th days after fertilization were not expected to occur as early as the first days of determination because the organic fertilizer material must undergo the mineralization process and was not observed in other studies when organic matter was used (Ribas et al., 2010; Ruijter et al., 2010; Almeida, 2012; Shan et al., 2015). According to Almeida (2012), while the peaks of N-NH$_3$ volatilization in urea fertilization occurred after the first two days, the highest peaks from gliricidia legume fertilizer were between the second and fourth days. Furthermore, a delay of at least four days for the onset of volatilization was reported for organic fertilizers (Ruijter et al., 2010).

Observations from our experiments can be attributed to the varied size distribution of the crushed sunn hemp residue, which was necessary for the adequate distribution of the organic matter on the surface of the boxes that could have increased the decomposition rate and release of N and other nutrients. In addition, the composition of crushed leaves and branches was not uniform, and could have different rates of decomposition. The finest materials, composed of leaves and small branches, decompose faster than that composed of thicker branches. This condition favored decomposition rates and differentiated ammonia volatilization times, causing the observed biphasic peaks. The first peak can be attributed to the decomposition of the finest material, followed by the larger components (Janzen & McGinn, 1991), which were degraded over a longer period of time.

Other factors may have contributed to the observed results in addition those already described, including high temperatures in the protected growth environment. The maximum temperatures within the protected environment were often higher than 30°C, with average temperatures generally above 25°C. Maximum or minimum temperatures and periodic thermal variations affect the velocity of the reactions occurring in the soil and microbial activity (Wolf & Wagner, 2005). At temperatures close to 28°C, microbial reactions are maximized, while at temperatures below 25°C and above 35°C there is a sharp decrease in the microbial reactions (Moreira & Siqueira, 2006).

Although we do not have data on pH variation over time, it is known that green manure can raise pH of the soil due to the accumulation of basic reaction cations in green manure (Perin et al., 2010). High soil pH promotes the conversion of NH$_4^+$ into NH$_3$ (Shan et al., 2015), which partially explain the results.

The application of AS in addition to the application of sunn hemp fertilizer may have also contributed to the first volatilization peaks. Although there were no mineral fertilizer applications inside the volatilization chambers, their dilution in the irrigation water may have contributed to a priming effect, that is, in addition to the decaying legume material inside the chambers, decomposition of the organic matter of the fertilizer in the soil may have also occurred (Alfaia 1997).

After 123 days, accumulated volatilization of N-NH$_3$ was 41.88 kg ha^{-1} N (Figure 2); nine days later, the value was 20.83 kg ha^{-1} N, which corresponded to almost 50% of the total volatilized N. Studies carried out in Viçosa, in the state of Minas Gerais, showed that the half-life of N in decomposing sunn hemp residues was less than 15 days (Perin et al., 2006; Diniz et al., 2014); whereas in the labile compartment, it is 1.8 days compared to a dose equivalent to 3 t ha^{-1} (Diniz et al., 2014). These results indicate that sunn hemp has a potential for rapid mineralization, especially under temperature and irrigation conditions similar to those in the present study. These results further suggest potential losses by N-NH$_3$ volatilization, which could explain part of the results of the present work.

The volatilized N may come from the legume-based fertilizer, from the mineral fertilizer used to supplement the irrigation water, and from the organic matter native to the soil. The amount of N volatilized after 30 days corresponds to 10.7% of the total N applied (285 kg ha^{-1}), which is considerably low compared to the values observed in urea fertilizer use, which can be close to 40% of the total N (Soares et al., 2012). Similar studies with *Mucuna cinerea* and gliciridia indicated N-NH$_3$ losses of 6.13% and 11.51% of

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the total N applied after 30 and 23 days (Ribas et al., 2010; Almeida, 2012), respectively; these values are very close to the N losses measured in the present study.
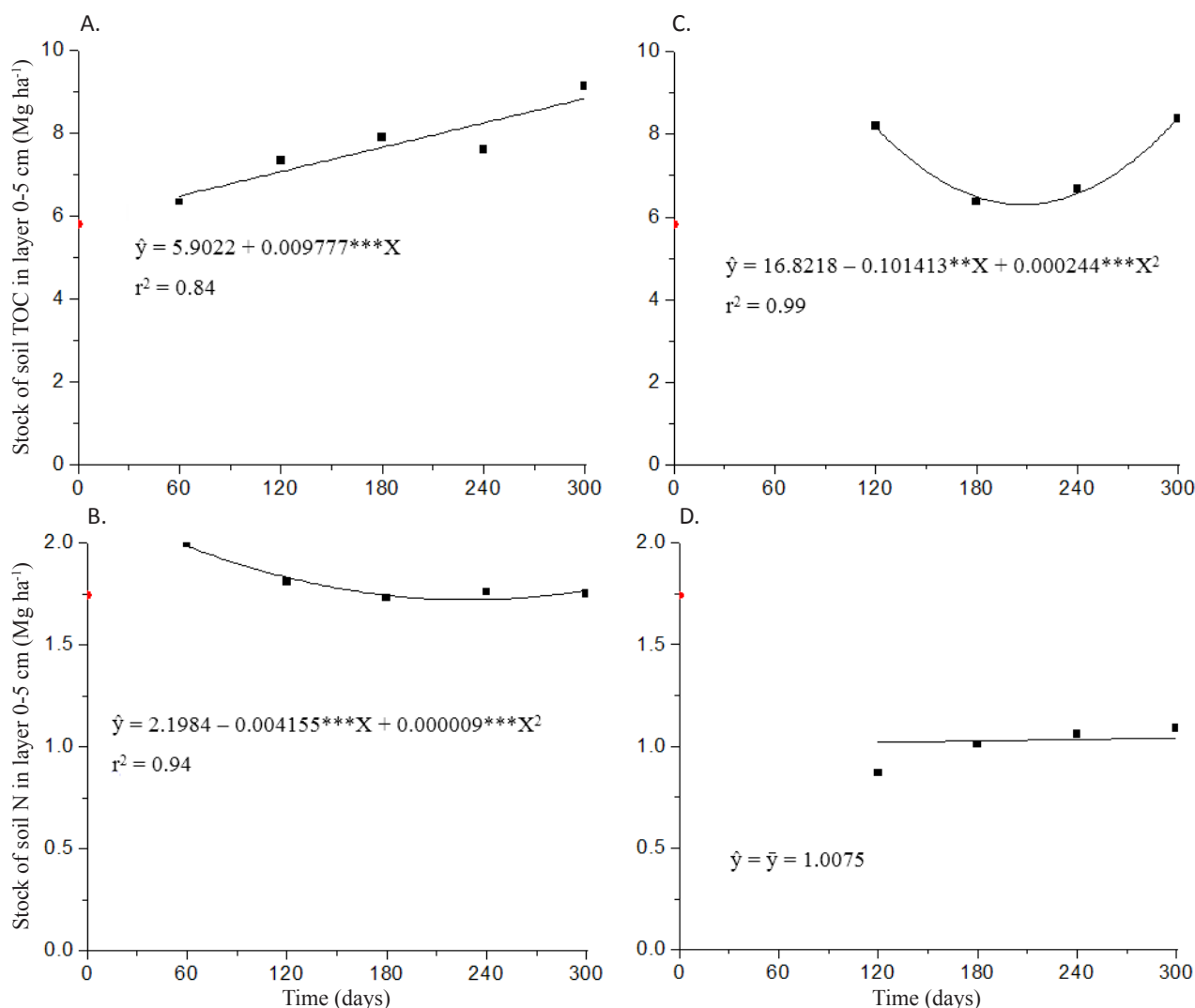
TOC and TN stocks

Differentiated behaviors were observed for TOC and TN stocks at both depths. There was an increased linear response of TOC over time in the 0–5-cm layer, measuing 5.81 Mg ha^{-1} at time zero to 8.84 Mg ha^{-1} at 300 days (Figure 3A). On the other hand, TN increased from 1.74 to 1.98 Mg ha^{-1} for 60 days, and then presented a quadratic behavior with a decrease in values at 231 days, when it presented a stock of 1.72 Mg ha^{-1} (Figure 3B). Regardless of the quadratic behavior with values that decreased from 120 days, the TOC stock increased in relation to time zero in the 5–10-cm layer at all other time points, starting from 5.81 Mg ha^{-1} in time (Fig. 3C), respectively, at 120, 180, 240, and 300 days, respectively. The TN stock decreased after 60 days compared to time zero (Figure 3D) but was not significantly time dependent.

The effects of maintaining residues on the soil surface and the consequences on soil organic matter are known in the literature, even with considering the subsurface layer (Carmo et al., 2012; Gauder et al., 2016). Generally, this response demands a longer period of time, such as a few years (Carmo et al., 2012). For our results, some factors may have contributed to faster replenishment of TOC stocks due to green manuring in soil both layers. Among them, we suggest that the high temperatures in the protected environment, the constant irrigation, and the soil kept at field capacity, as well as the chemical fertilizations.

The addition of organic matter to the soil surface may have led to the rapid incorporation of C from the organic matter into deeper soil layers, enriching these layers with organic C (Poirier et al., 2014; Gauder et al., 2016). The periodic irrigation of the cultivation boxes in the experiment may have favored the decrease and stabilization of C and N soluble forms (Neu et al., 2011) in deeper soil layers, or even in the organic form (small residues). Because of the biological activity and the decomposition of these residues, C and N bound to the organic fractions or in their mineral forms, could move to the deeper soil layers and promote the initial reduction of TOC and non-alteration of N, which

A.

$\hat{y} = 5.9022 + 0.009777^{***}X$

$r^2 = 0.84$

C.

$\hat{y} = 16.8218 - 0.101413^{**}X + 0.000244^{***}X^2$

$r^2 = 0.99$

B.

$\hat{y} = 2.1984 - 0.004155^{***}X + 0.000009^{***}X^2$

$r^2 = 0.94$

D.

$\hat{y} = \bar{y} = 1.0075$

*** and ** indicate significant differences of 0.1% and 1%, respectively.

Figure 3. TOC and TN soil stocks at 0–5 cm and 5–10 cm depths after *C. juncea* mass application.

is much more mobile, with little accumulation in one layer or another. In the deeper soil layers, the organic C contents were lower and the retention of the organic material from the decomposing residues could be favored (Poirier et al., 2014). This may have also contributed to a higher relative carbon increment in the 5–10-cm layer after 120 days. The behavior of the organic matter residues and their effect on C and N stocks can be quantified from the TN because the decrease or increase of the stocks might be associated with the mineralization of organic N sources in the soil and its consequent absorption by the coffee plants, leaching to deeper soil layers, and/or volatilization losses.

Stocks of TN at time zero in the 0–5-cm soil layer were equivalent to 1.74 Mg ha^{-1}. Then, approximately 285 kg ha^{-1} N were added via AS and sunn hemp dry matter. After 60 days, approximately 36.10 kg ha^{-1} N was lost by N-NH$_3$ volatilization, and the approximate TN stock in the first soil layer was 1.98 Mg ha^{-1}. There was an increase of 0.24 Mg ha^{-1} in the 0–5-cm layer. At 120 days, N-NH$_3$ volatilization losses reached approximately 41.68 kg ha^{-1} and the TN stocks in the soil were 1.83 Mg ha^{-1}, which resulted in an increment of 90 kg ha^{-1} N in the 0–5-cm soil layer in relation to time zero. We observed differences of 8.90 and 153.32 kg ha^{-1} N at 60 and 120 days, respectively, from the initial stocks, the N input via fertilizers, the ammonia volatilization losses, and the final stocks. These differences indicate the absorption of N by the coffee plants, leaching to deeper soil layers that were not measured, and/or losses by other volatile forms. However, it is possible that there was still N in the mass of the decomposing sunn hemp in that period that had not been released into the soil and was, therefore, not included in our analysis.

The variation in soil TN stocks in the 0–5-cm layer was positive in relation to time zero at 180 days (Figure 3B). In the 240-day evaluation, the variations in soil TN stocks fluctuated between negative and positive. The responses after 240 days occurred by applying part of the nitrogen fertilization of the first year after planting, when the equivalent of 203.4 kg ha^{-1} of N in the form of AS was supplied.

No time dependency was observed in the N stocks in soil from the 5–10-cm layer; they were negative in relation to the average variations at time zero (Figure 3D). This indicated N mineralization in the organic compartments and N output from this layer, that occurred mainly from nutrient leaching and absorption by the coffee plants.

In the topmost layer, we observed a constant decomposition and mineralization of the sunn hemp organic matter, which certainly determined the results, even with mineralization of the organic compartments, N absorption by the coffee plants, possible leaching, and losses by volatilization.

Conclusions

From this experiment, we conclude that N-NH$_3$ volatilization is rapid and the highest N-NH$_3$ volatilization occurs soon after fertilization of the coffee trees with sunn hemp and AS.

The observed N-NH$_3$ volatilization is fast, but can be considered low.

The rapid volatilization of N-NH$_3$ indicates rapid decomposition and mineralization of the organic residues from the legume-based green manure, which was reflected in the rapid increase of TN and TOC stocks in relation to time zero, with the exception of NT stocks in the 5–10-cm soil layer.

The increase in TOC in the two soil layers was not followed by the increase in TN stocks, indicating that the N had other fates rather than accumulation in these layers.

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