Organic Nitrogen Mineralization Rate in Sewage Sludge Amended Mine Soil

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Abstract

The concept and importance of soil quality is pertinent to reclaimed mine-soils and other disturbed ecosystems. Adding organic amendments has been used as a means for ameliorating mine soils and improving their quality. This study was carried out on lignite mine tailings at Meirama (North-western Spain). The spoils are heterogeneous and mainly consist of a mineral fraction (quartz, feldspar and mica) and an organic fraction (lignite rests). A laboratory experiment was conducted to investigate the effects of amending lignite mine spoil with three different surface treatments: a control (nothing added), and municipal sewage sludge (SS) at rates of 5, 25 y 50 Mg ha^{-1}. Triplicate samples of soil and each treatment were incubated from 0 to 90 days at constant moisture and temperature by aerobic process. Each sewage sludge dose was packed into PVC columns and inserted vertically into the recipient containing the upper layer of mine-soil. A set of the incubated columns was removed at 15 days intervals and the soil underlying each column was sampled to analysis. Total organic, inorganic, NO_3^- and NH_4^+- N, N mineralization rate were measured.

The addition of sewage sludge increases the soils inorganic nitrogen content. Sewage sludge application produced an immediate increase of the inorganic N at all dosage levels, mainly in ammonium form. The N mineralization in the amended soil was higher than in the control soil, mainly in the case of those treated with 25 Mg ha^{-1}. With the higher dosage of sewage sludge, immobilization of N occurs due to the addition of a large quantity of organic matter, of which 45% consists of fractions resistant to degradation in this short period. The net mineralised organic N was always positive, except at the end of the period of incubation in the 5 Mg ha^{-1}sewage sludge treated soil. The highest values were presented in the soil treated with the 25 Mg ha^{-1} sewage sludge dose.

The nitrogen mineralization rate, calculated as a decrease in the organic nitrogen content, overvalues the amount of inorganic nitrogen that is available.

The measurement of the cumulative amount of NH_4^+-N and NO_3^--N provided a much lower estimation of N mineralization under incubation conditions. The underestimation of N mineralization might be caused by loss of N through denitrification and ammonium volatilisation.

Introduction

Soil quality research has focused on intensively-managed agricultural and forest soils, but the concept and importance of soil quality is pertinent to disturbed systems such as reclaimed mining soils. The restoration of soil function and mining soil quality is essential to long-term ecosystem stability (Bendfeldt, 2001). Restoration of mining soil quality and ecosystem function of disturbed sites depends on reclamation that allows reestablishment of vegetation that can thrive and sustain itself. Seaker and Sopper (1988) stressed the importance of soil organic matter accumulation, decomposition, and minimum levels of organic C and N contents for productive mine soils. Most opencast mining sites lack organic matter levels necessary for optimum soil functioning.

The depauperate nature of mining soils may benefit from addition of organic amendments that will initiate nutrient cycling and help overcome other chemical and physical limitations (Sengupta 1993, Tennessee Valley Authority 1963). Organic amendments applied on opencast mining soils would accelerate nutrient cycling, provide a receptive environment for vegetation, and improve overall soil quality and productivity. Reclaiming these highly disturbed soils is an important process in returning land to a stable state.

Topsoil is an important aspect in mine-spoils reclamation. Mining soils have lower rates of decomposition, decreased nitrification, and reduced enzyme activity (Lindeman et al., 1984). Organic matter is the food source for soil microorganisms and serves as a slow-release nutrient source for plants. Slowed decomposition hinders the release of essential nutrients and results in a nutrient sink. Sewage sludge has been used frequently as an organic amendment for soils (Clapp et al,. 1986). Sewage sludge is a residual product from the treatment of municipal wastewater (Linden et al., 1995).

Sewage sludge contains essential plant nutrients, primarily Nitrogen and Phosphorus. The organic matter in sludge is a key component to its success as an organic amendment. Sewage sludge is a convenient amendment because it has many fertilizer constituents in one application.

Restoring Nitrogen cycling is one of the major goals of ecosystem recovery of soils overlying opencast mining sites, whether they are returned to natural habitats or agricultural enterprises. Because opposing N transformations occur simultaneously, estimates of N cycling processes based on net changes of N substrates and products are unlikely to provide a clear indication of the impact of soil restoration practices at sites disturbed by surface mining.

The specific objective of this study was to determine the ability of sewage sludge amendment to improve mine soil quality as measured by N mineralization and the fate of mineralised N as a potential leachate from the sewage sludge using a laboratory incubation approach.

Material and methods

This study was carried out on lignite mine tailings at Meirama (North-western Spain, 42º12'N, 8º26W). The spoils are heterogeneous and mainly consist (Leirós et al., 1989) of a mineral fraction: quartz, feldspar, mica kaolin and, in minor proportion, illite, vermiculite and smectite; and an organic fraction (highly stabilized and biodegradable with difficulty): lignite rests (Seoane et al., 19925).

Mine-spoil samples, for the incubation experiment, were collected from the top 30 cm. Aerobic digested sewage sludge is a residual product from the treatment of municipal wastewater from Vigo (Spain).

Some significant properties of mine-spoil and sludge are presented in Table 1.

A laboratory experiment was conducted to investigate the effects of amending lignite mine spoil with three different surface treatments: a control (nothing added), and municipal sewage sludge at rates of 5, 25 y 50 Mg ha^{-1} (T-1, T-2 and T-3 respectively).

Nine replicate samples of spoil and each treatment were incubated from 0 to 90 days at constant moisture and temperature by aerobic process. Each sewage sludge dose was packed into PVC columns (5 cm diameter and 10 cm height columns) and, each one, inserted vertically into a recipient containing the upper layer of mining soil, according to the method proposed by (He et al., 2000). The moisture content of the sewage sludges was adjusted to their field water holding capacity before being packed into the column. Moisture equilibrium between the incubated sample inside the column and the soil was attained through the bottom and four side holes (6 mm diameter), which were made evenly at the middle of the column. The bottom and the four side holes were covered with nylon screen (400 mesh) for separating the sewage sludge sample from the surrounding soil.

The nine replicate samples of spoil and each treatment were introduced in a chamber under controlled conditions (85 per cent air relative humidity and 25ºC). Three columns from each treatment were sampled at 15-day intervals to measure total-N, NH_4^+-N and NO_3^--N concentrations in the sewage sludge. A soil core (10 cm depth, 5 cm diam.) was also sampled directly beneath each sewage sludge column and analyzed for total-N, NH_4^+-N and NO_3-N.

Three different procedures were used to determine N mineralization rates of sewage sludge (He et al., 200): 1) The cumulative amount of NH_4^+-N and NO_3^--N in the sewage sludge sample in the PVC column, 2) the cumulative amount of NH_4^+-N and NO_3^--N in both the sewage sludge sample (in the PVC column) and the soil directly beneath the sample and 3) the difference in the organic concentration of the sewage sludge sample before and after incubation.

Subsamples of the sewage sludge packed into the columns were analysed for moisture content and $CaCl_2$-extractable NH_4^+-N and NO_3^--N. The increase in NH_4^+-N and NO_3^--N in the soil below the column over the control was considered to be due to leaching from the sewage sludge after mineralization. The amounts of NH_4^+-N or NO_3^--N leached into the soil were calculated from the increased concentration of NH_4^+-N or NO_3^--N and the total weight of the soil core sampled. This amount was added to the N forms measured in the sewage sludge to determine total plant-available mineral N for an experimental period. At the end of an incubation period, the dry weight of residual sewage sludge was measured and concentrations of total C and N and mineral N in the residual sewage sludge were determined to estimate residual organic-N. The difference in organic-N before and after incubation in these sewage sludge samples provided an estimate of N mineralised during this experimental period.

$CaCl_2$-extractable NH_4^+-N and NO_3^--N in both sewage sludge and soil were determined by shaking, for 2 hours, a dried (at 40ºC) sample at a 1:10(w/v) ratio with 0.01M $CaCl_2$ at 20ºC (Houba et al., 2000). The suspension is centrifuged for at least 10 minutes at about 1800 g. A drawn aliquot of the supernatant is acidified with HCl 1M and analysing concentration of NH_4^+-N and NO_3^--N with a segmented-flow auto analyser (Bran Luebbe-AA3).

The nitrogen mineralization rate (NMR) of the sewage sludge under experimental conditions was calculated according to He et al. (2000):

$$NMR\% = 100\left[\frac{\text{Org. N before inc.} - \text{Org. N after inc.}}{\text{Org. N before inc.}}\right]$$

where:

organic N=total N–mineral N (NH_4^+-N+NO_3^--N)

Unrecovered mineral N = total mineral N – recovered mineral N

where:

total mineral N = mineral N from organic N mineralization, estimated by decrease in organic N during the incubation.

Recovered mineral N = mineral N in the sewage sludge + mineral N in the soil below as determined by the Ca Cl_2 extraction method.

Statistical analysis of the mineralization rate and other data was performed using the ANOVA procedure and means separated using contrast statements and least significant differences (LSD) (SAS Institute, Inc. 1996).

Results and discusión

Dynamics of the transformation and mineralization of Nitrogen

The residual sludge contains 23.8 g. Kg^{-1} of N, principally in an organic form (Table 1) and the inorganic N is fundamentally in the form of NH_4^+. The evolution and mineralization of N differs between the different doses of sludge (Figure1). The Nitrogen mineralization rate of the sludge inside the columns was relatively quick, above all in that which contained the dosage of 5 $Mgha^{-1}$ in which a rapid transformation of the organic-N was produced. Thus, at 15 and 45 days of incubation, 29 and 74% respectively of organic-N had been mineralised.

Table 1. Some properties of minesoil and sewage sludge

	Sewage sludge	Minesoil	
pH_{H20}	7.93	4.91	
O. M. ($g\ kg^{-1}$)†	366.6	15.93	
Total N ($g\ kg^{-1}$)	23.8	0.85	
C:N ratio	7.7	10.89	
Organic-N ($g\ kg^{-1}$)	21.01	0.84	
NH_4^+ - N ($mg\ kg^{-1}$)	2780	5.0	
NO_2^- - N ($mg\ kg^{-1}$)	1.2	u.l.	
NO_3^- - N ($mg\ kg^{-1}$)	1.5	4.4	
P ($mg\ kg^{-1}$)	55.12	u.l. ‡	
Total content ($mg\ kg^{-1}$)			
Cd	0.20	1.80	
Cu	0.46	12.38	
Cr	0.07	13.88	
Fe	28.05	17802.13	
Mn	3.2	207.58	
Ni	4.1	14.039	
Pb	1.7	u.l. ‡	
Al	81,14	49342.76	
Total content of Na, Ca, K, Mg and Al ($mg\ kg^{-1}$)		Exchangeable cations and cationic exchange capacity ($cmol\ kg^{-1}$)	
Na	113	Na	0.22
Ca	278	Ca	0.84
K	494	K	0.73
Mg	941	Mg	0.66
		Al	0.32
		H^+	16.33
		$CEC_{(+)}$	4.94

†: Organic matter, ‡: undetectable level

During the initial stage of incubation, NH_4^+-N is the predominant form of inorganic-N in the sludges and soils. It increases rapidly in the sludge and soil, above all in the lower the dosage rates, reaching a maximum at between 15 and 45 days of incubation, decreasing afterwards at the end of the incubation period. The decrease of the NH_4^+ was generally accompanied by an increase in $N\text{-}NO_3^-$, indicating that the NH_4^+ liberated in the mineralization of the sludge was nitrified to $N\text{-}NO_3^-$.

Nitrogen mineralization rate

Under incubation conditions, the measurement of the difference in organic-N content before and after incubation provides very high mineralization rates for all the dosages of sludge employed (Figure 2). The values of mineral-N measured in the soil and sludge after incubation, extracting NH_4^+ and $N\text{-}NO_3^-$ from the soil and sludge, indicates a much lower rate of mineralization. The measurement of the accumulated amounts of NH_4^+ and $N\text{-}NO_3^-$ in the sludge shows a much lower estimation of the mineralization rate when compared with the other two procedures.

The mineralization of N is frequently determined by the calculation of the accumulated amount of NH_4^+ and $N\text{-}NO_3^-$ in periodic samplings (Garau et al., 1986; Simms, 1995). This does not take into account possible losses by volatilisation and lixiviation and sub-estimates the rate of mineralization. The decrease of organic-N in the sludge during incubation is a clear indication of mineralised-N although it overestimates the amount mineralised. However, a part of the N in the sludge that was mineralised is not determined in a mineral form by extraction because losses have been produced during incubation. The amount of mineral-N available for the plants is generally less than that estimated by this procedure.

The measurement of the extractable NH_4^+ and $N\text{-}NO_3^-$ in the soil directly underneath the columns containing the sludge provides a basis for estimating the amount of mineralised N that is lixiviated from these columns.

The control presents a negative rate of mineralization during the whole period of study, due to the immobilisation of the N. This is probably due to the growth of the microbial population under incubation conditions that use the scarce amount of existing inorganic-N to form their cellular structures. This causes an increase of organic-N and simultaneously decreases the content available in an inorganic form.

It has been shown that the amount of mineralised organic-N differs from that of inorganic-N ($N\text{-}NH_4^+ + N\text{-}NO_3^-$) obtained by extraction with $CaCl_2$ from the sludge inside the columns and the soil below. This is due to the losses produced during the incubation process by the volatilisation of ammonium and denitrification of the nitrates.

For this reason, the mineralization rate, calculated as a decrease in the organic-N content, overvalues the amount of inorganic-N that is really available, which demands knowledge of the amount of N that is not recovered, but, however, has been mineralised. In order to do this, the method proposed by He et al. (2000) - based on the difference between the content of total-N and that recovered - has been used, both in the sludge and in the soil.

As can be seen in Figure 3, the amount of unrecovered N is higher in the case of treatment T-1 (5 $Mgha^{-1}$) than in the control, than in T-2 and in T-3 (25 y 50 Mg ha^{-1}). Due to the high mineralization rate produced with the dosage of 5 Mg ha^{-1}, the losses of N reach, at the end of incubation, 98% of the N that is mineralised.

The amount of unrecovered N is significantly different between treatment T-1 and the others, whilst these differences do not appear between the control and other treatments.

Negative amounts of recovered-N have been obtained in the control throughout the whole of the incubation period and with the dosages of 25 and 50 Mg ha^{-1} during the first 45 days. This appears to indicate that the amount recovered exceeds the total content of N. However, this is because the method of calculation used does not take into account the mineralization of the organic-N of the soil itself, which is produced under incubation conditions. After 45 days of incubation, and with the highest doses, losses of inorganic-N have been obtained, which have increased as the amount of sludge increased.

The amount of unrecovered inorganic-N was much higher in the soil that received a dosage of 5 Mg ha^{-1} of sludge than in those that received 25 and 50 Mg ha^{-1}, as the rate of mineralization is higher at that dosage. Although mineralization is lower and slower with higher doses, the amount of unrecovered inorganic-N is much lower and these losses occur in the final phase of incubation. Therefore, it is better to use the highest doses in order to supply adequate amounts of available-N to the plants, which allows a more rapid and more complete recovery of the vegetation on the mine-soil.

Conclusions

The net mineralised organic-N is positive in the soil treated with a dosage of 5 Mg ha^{-1} of sewage sludge, except at the end of the incubation period. The highest rates are produced in the soil treated with a dosage of 25 Mg ha^{-1} of sewage sludge.

The mineralization rate of N calculated as a decrease of the inorganic nitrogen content overvalues the amount of actually available inorganic-N.

The determination of the amount of NH_4^+-N and NO_3-N produces a better estimate of the mineralization of N under incubation conditions.

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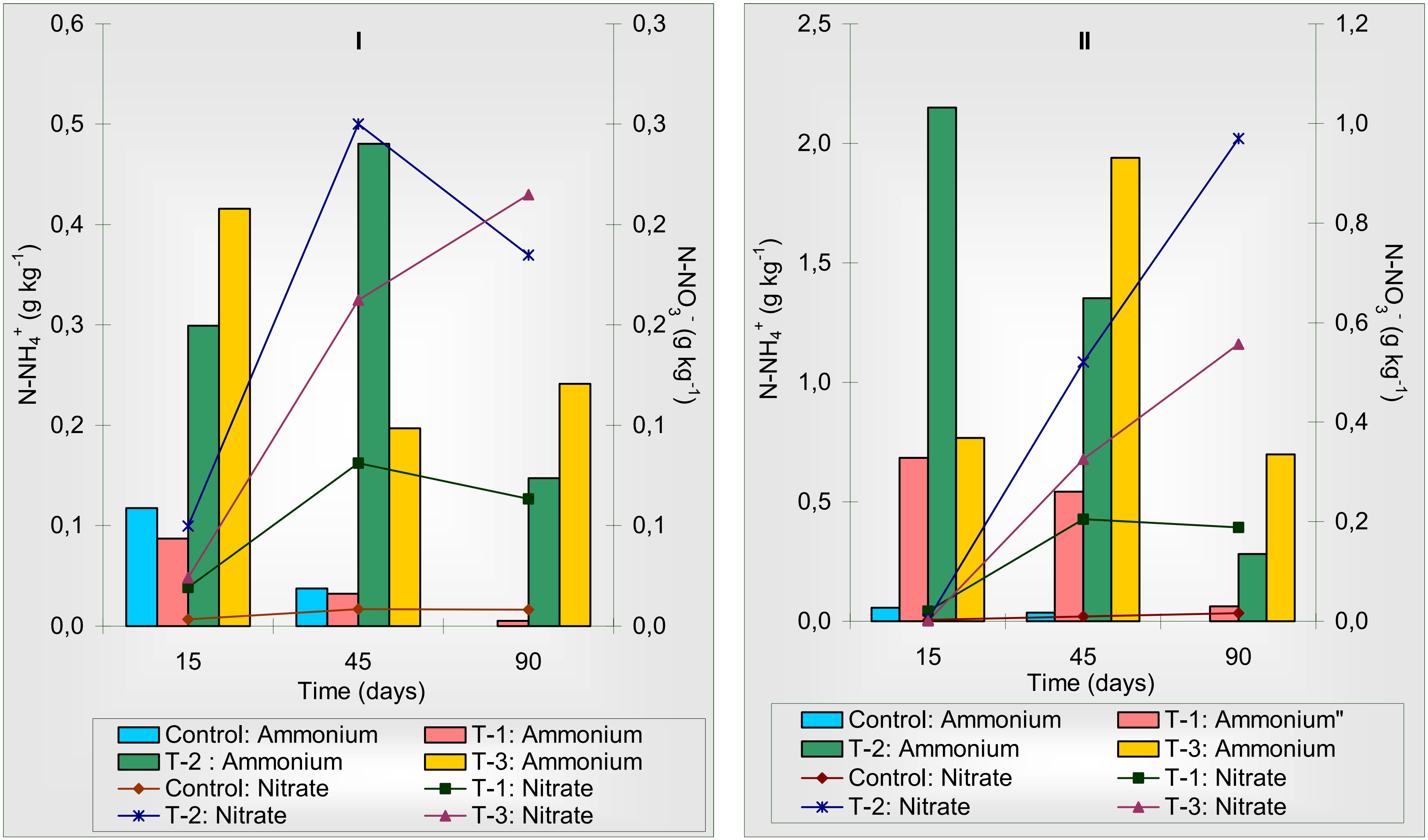

Figure 1. NH_4^+-N and NO_3^--N content variation: I. Soil directly beneath the PVC column. II Sewage sludge packed into the PVC column.

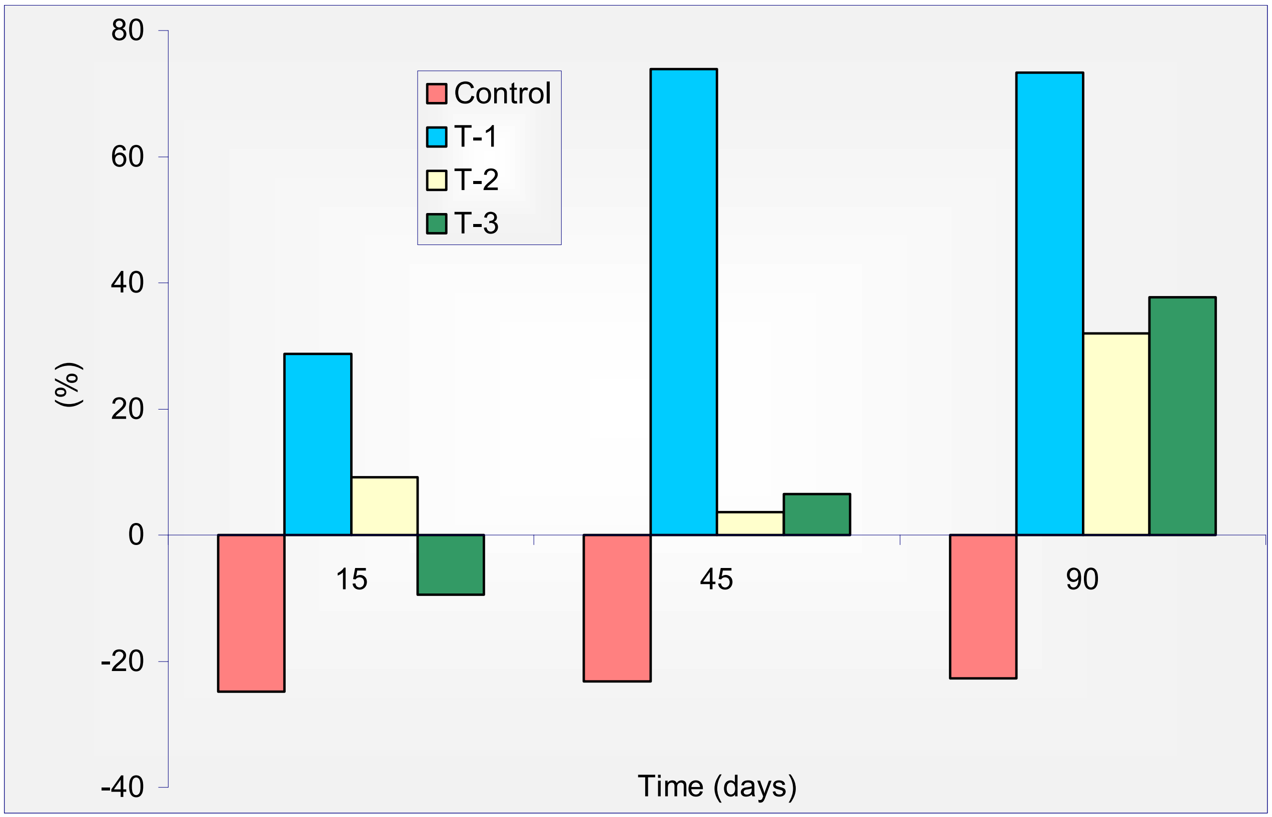

Figure 2. Nitrogen mineralization rate

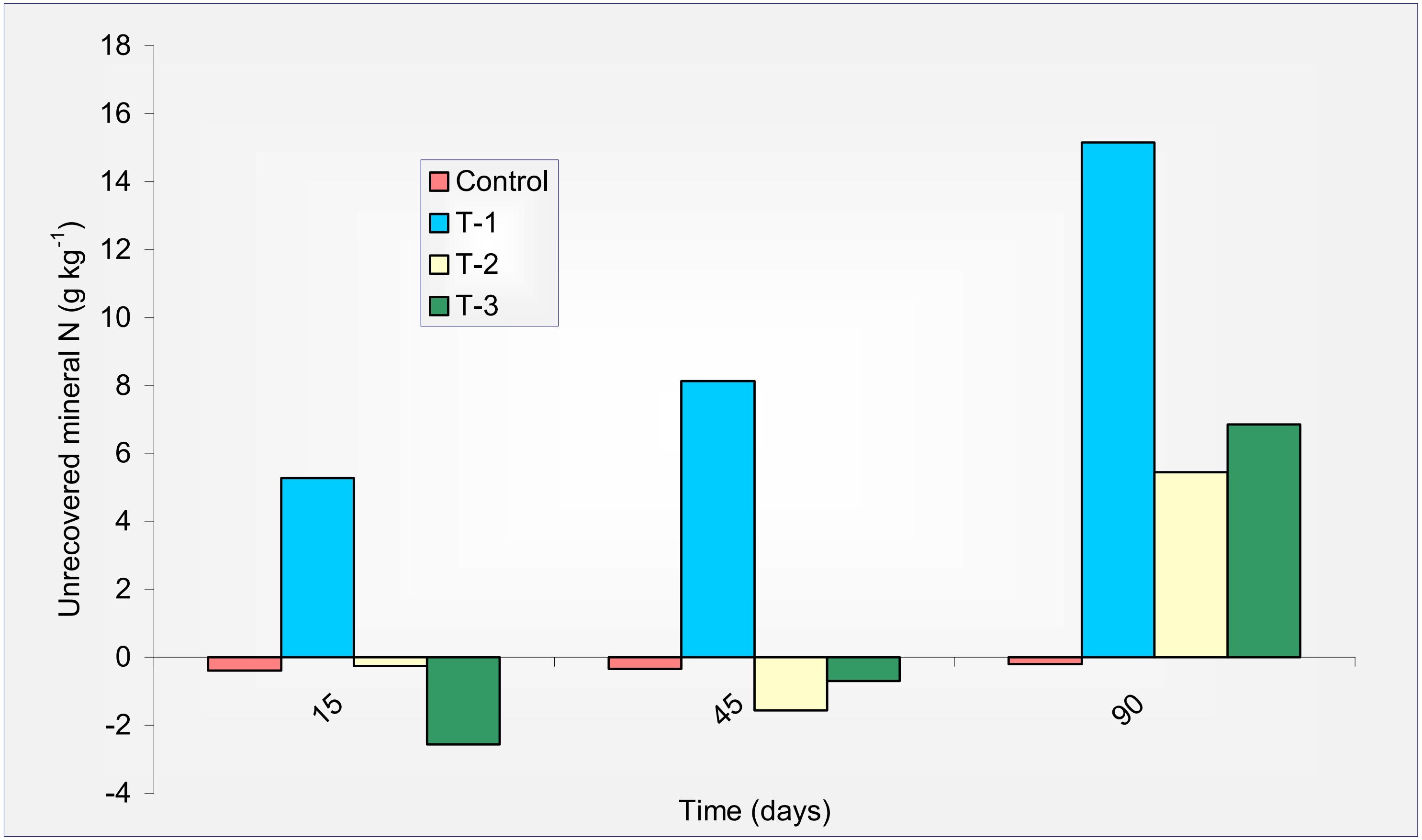

Figure 3. Unrecovered mineral-N.